THE SCALING-UP
OF
CHEMICAL PLANT & PROCESSES

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THE SCALING-UP OF CHEMICAL PLANT AND PROCESSES

INTRODUCTORY PAPER

By R. EDGEWORTH JOHNSTONE, M.Sc., M.I.Chem.E., F.R.I.C.* and
Prof. M. W. THRING, M.A., M.I.Chem.E.†

SUMMARY

When the scale of physical and chemical processes is changed, the results are sometimes unexpected. Deviations can often be evaluated and allowed for by means of the Principle of Similarity. There are two methods of deriving similarity criteria, by dimensional analysis and from differential equations. Where applicable the latter method is to be preferred. Extrapolation by means of a power law relation enables model and prototype to be compared under conditions that are not strictly similar. Where a mixed régime exists, reliable predictions of scaled-up performance may be impossible.

The objects of this introductory paper are to review briefly some of the problems associated with change of scale in chemical engineering, summarise past investigations in this field, mention a few general principles and empirical rules, and indicate one or two aspects of the subject which might repay further investigation.

Problems

It is well known that when a new chemical process is transferred from the laboratory to the pilot plant, unforeseen difficulties in operation are often met. Further technical difficulties can occur when the process is transfered from the pilot plant to full-scale operation. In each case the change of scale is liable to give rise to unexpected physical or chemical effects. It is not so generally realised that the reverse condition can occur, and that a process which fails to work in the pilot plant may proceed perfectly satisfactorily on the large scale. Hall, inventor of the electrolytic process for the manufacture of aluminium, never succeeded in making his original small cell function properly. After three years of failure he had the courage to build a large cell of 35 kw. capacity, and the difficulties that had dogged his small-scale experiments " disappeared as if by magic ".[1] Inventors and their sponsors seldom have as much confidence in their ideas as Hall had, and we shall never know how many potentially valuable processes have been abandoned at the pilot-plant stage because of some reverse scale effect which would have righted itself automatically in a larger plant.

So much for the problems of scaling-up: but the chemical engineer is also confronted from time to time with the necessity for scaling-down. It is often desired to know the effect of changed operating conditions or structural alterations on the performance of some piece of full-scale plant. If the plant is large and slow in its response to changes, then experiments on the full scale are likely to be very costly, especially if they involve an interruption of production or any spoilage of the product. Whenever possible it is much better to construct a model of the full-scale apparatus and carry out experiments with that. Such considerations apply especially to large furnaces, and furnace models are widely used to predict the effects of variations in shape or changes in operating conditions.[2]

A study of the effects of change of scale and of the factors governing scale effects will generally indicate the conditions under which small- and large-scale units will give comparable performances. Sometimes the scale effects are found to be so large and incalculable that there are no conditions under which the performance of a large-scale plant could reliably be predicted from that of a small model. In such cases the only safe procedure is to test a succession of pilot units of gradually increasing size. At the other extreme, the scale-up study may show that no scale effects whatever are to be anticipated. In that case it might well be feasible to omit the pilot-plant stage entirely and proceed directly from the laboratory to the full scale. The Shell hot-acid alkylation process described by McAllister[3] was one of the early processes to be thus scaled up in one step, and there have since been others, notably the isotope separation processes that were associated with the Manhattan Project.

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History

The basic principle governing the effects of change of scale on physical and chemical processes is the Principle of Similarity, enunciated by Isaac Newton in relation to systems composed of solid particles.[4] Its first practical applications were in the field of hydrodynamics, and towards the end of the last century W. Froude was using similarity theory to predict the wave drag on ships' hulls from experiments with models, while Osborne Reynolds employed models of river beds and estuaries to simulate erosion effects. A more recent application has been for the prediction of aircraft performance from experiments with models.

In the field of chemical engineering the application of similarity principles to geometrically similar systems appears to have been first used to correlate the performance of liquid mixers, a phenomenon not yet susceptible to exact mathematical analysis. Early studies were carried out by Hixson and his co-workers,[5] and more recent work has been done by Rushton.[6] Similarity theory forms the basis of the commonly-used dimensionless equations for heat and mass transfer on the pattern of the Dittus-Boelter equation. Procedures have recently been given for the direct scaling up of heat transfer coefficients,[7] and several workers have applied the principle of similarity to the study of crushing and grinding.[8]

Chemical reactions are more complex than the physical processes involved in most chemical engineering operations, since in a chemical reactor there are fluid dynamic, thermal, and chemical effects to be taken into account. The principle of similarity was first applied to the scaling up of chemical reactions by Damköhler,[9] and its application in this field has since been extended by others.[10]

Principles

In its simplest application the Principle of Similarity attempts to represent a physical or chemical process by an unspecified relation between several dimensionless groups, one of which contains the unknown variable. If the groups containing the known variables are caused to have the same value on the small- and large-scale, then the group containing the unknown variable will also have the same value. In this form the principle of similarity pre-supposes that the systems to be compared are geometrically similar.

Geometrical similarity in the sense called for here does not necessarily require the small apparatus to be an exact miniature of the large one. In general, geometrical similarity to a given full-sized prototype may be attained by three different types of smaller apparatus, namely :

(1) a *model*, *i.e.* a complete scale model of the working portion of the prototype,

(2) an *element*, *i.e.* a full-sized replica of a portion of the prototype,

(3) a *model element*, *i.e.* a scale model of an element.

Models and model elements may be either regular or distorted. A distorted model has different scale ratios along different co-ordinates. Thus there is a considerable latitude in the shape of small-scale unit that may represent a given prototype while conforming to the requirement of geometrical similarity.

Where the form of the equation governing a physical or chemical process is entirely unknown, the appropriate dimensionless groups can in theory be derived by dimensional analysis using either Rayleigh's method of indices[11] or Bridgman's method of matrices[12]—always assuming that all the physical variables which significantly affect the process are known. This last assumption is the weak point of the dimensional analysis technique, since where the equation governing the process is unknown it is not clear how one can be sure of selecting the correct variables. If only one relevant variable is omitted the results of dimensional analysis can be entirely misleading.

There is another, and in the authors' view a better, way of deriving dimensionless groups. For nearly all of the physical and chemical processes of interest to chemical engineers the fundamental differential equations are known, although in many cases they cannot be integrated. Provided these differential equations are dimensionally homogeneous (as all complete physical equations should be) they can be simplified to a generalized dimensional form by dropping the differential signs and substituting for linear co-ordinates a generalized linear dimension L. Dividing across by one term then gives the relevant dimensionless groups.

When dimensionless groups are thus derived from a complete set of differential equations there is no doubt as to whether the correct variables have been included.

For example, the fundamental differential equations for the isothermal flow of a Newtonian viscous fluid are the Navier-Stokes equations :

$$\rho\frac{\partial u}{\partial t}+\rho\left\{u\frac{\partial u}{\partial x}+v\frac{\partial u}{\partial y}+w\frac{\partial u}{\partial z}\right\}=\rho\, g\cos\alpha_x-\frac{\partial p}{\partial x}$$
$$\tfrac{1}{3}\frac{\partial}{\partial x}\left\{\frac{\partial u}{\partial x}+\frac{\partial v}{\partial y}+\frac{\partial w}{\partial z}\right\}+\mu\left\{\frac{\partial^2 u}{\partial x^2}+\frac{\partial^2 u}{\partial y^2}+\frac{\partial^2 u}{\partial z^2}\right\} \quad . \quad . \quad . \quad (1)$$

a formidable enough expression, with two more corresponding equations for the y and z axes. Simplifying this down to one generalized dimensional equation :

$$\left[\frac{\rho v}{t}\right]+\left[\frac{\rho v^2}{L}\right]=\left[\rho g\right]-\left[\frac{\Delta p}{L}\right]+\left[\frac{\mu v}{L^2}\right]$$

Dividing across by $\rho v^2/L$

$$\left[\frac{L}{vt}\right]+1=\left[\frac{Lg}{v^2}\right]-\left[\frac{\Delta p}{\rho v^2}\right]+\left[\frac{\mu}{\rho v L}\right]$$

For steady flow L/vt drops out. Rearranging and omitting the plus and minus signs (which apply only to the differential form) :

$$\frac{\Delta p}{\rho v^2}=\phi\left(\frac{\rho v L}{\mu},\frac{v^2}{Lg}\right) \quad . \quad . \quad (2)$$

This is the familiar generalised dimensionless equation for fluid flow. It represents the pressure coefficient as an unspecified function of the Reynolds and Froude groups. In geometrically similar flow systems, provided the Reynolds and Froude groups are both equal, the pressure coefficients will also be equal. Where the flow systems are closed and filled with a single fluid the effect of the Froude group is negligible and the relation reduces to one between the pressure coefficient and Reynolds number. In cases of wave or vortex formation in a mobile fluid having a free surface the effect of Reynolds number may be small.

The strict Principle of Similarity requires geometrically similar systems to be compared at equal values of the appropriate dimensionless groups called Similarity Criteria. In the past this requirement has limited the application of the principle to chemical engineering plant, since in many cases it is either impracticable or impossible to achieve. For example, in a fully-baffled paddle mixer the requirement of equal Reynolds numbers means that the r.p.m. must vary inversely as the square of paddle diameter. For large scale-up factors this would mean either an uneconomically slow paddle speed in the large-scale mixer or a speed in the model so high that cavitation would occur. The model would, in fact, consume considerably more power than the large mixer.

To meet this difficulty the method of extrapolation has been proposed.[13] It has been found empirically that the unspecified function of the generalized dimensionless equation can in many cases be quite well represented by a power function, *e.g.* for closed fluid-flow systems :

$$\frac{\Delta p}{\rho v^2}=\text{Const.}\left(\frac{\rho v L}{\mu}\right)^x \quad . \quad . \quad (3)$$

The method of extrapolation is to :

(1) measure the performance of the model at a convenient speed,

(2) predict the performance of the large-scale apparatus at an equal value of the similarity criterion,

(3) extrapolate this performance to a different value of the similarity criterion assuming a power law relation with an exponent either determined experimentally or taken from the literature.

For systems in which the same fluids are used on the small and large scale the extrapolation process can be reduced to a simple formula. For example, in a fully-baffled paddle mixer the generalised dimensionless equation in terms of power consumption is :

$$\frac{Pg}{\rho N^3 d^5}=\text{Const.}\left(\frac{N^2 d\rho}{\mu}\right)^m \quad . \quad (4)$$

Or, where fluid properties are constant,

$$P \propto N^{3+2m} d^{5+m}$$

Letting primed symbols represent values in the large-scale mixer and unprimed symbols values in the model, the ratio of power consumption is given by :

$$\frac{P'}{P}=\left(\frac{N'}{N}\right)^{3+2m}\left(\frac{d'}{d}\right)^{5+m} \quad . \quad . \quad (5)$$

For geometrically similar mixers d'/d is the linear scale-up ratio. Equation (5) enables the power consumption of the large mixer at any paddle speed to be predicted from that of the model at any paddle speed provided the value of m is known. Under conditions of vigorous agitation it has been found experimentally that m is almost zero. The scale relation then becomes very nearly :

$$\frac{P'}{P} = \left(\frac{N'}{N}\right)^3 \left(\frac{d'}{d}\right)^5 \qquad . \qquad . \qquad (6)$$

In equations (5) and (6) it is assumed that the same fluid mixture at the same temperature is being stirred in both mixers.

Whether or not the fundamental differential equations governing a process are known, it is essential to discover the rate-determining factor or régime of the process before any prediction can be made about the effects of change of scale.[14] The three broad types of régime that are met with in chemical engineering are dynamic, thermal, and chemical. The prevailing régime must no be confused with the nature of the process itself. For example, heat transfer by forced convection is a thermal process, yet it takes place under a dynamic régime because the rate of heat transfer is a function of the fluid velocity. Similarly a chemical reaction, the rate of which is limited only by the rate at which the reactants can be brought together by mixing, is subject to a dynamic régime. Natural convection, on the other hand, is a thermal process subject to a thermal régime, since here the rate-determining factor is the temperature difference.

In the absence of other knowledge there are empirical criteria by which certain types of régime may be distinguished. Two of the most important are the temperature coefficient and the effect of fluid velocity or rate of stirring. Most physical processes are subject to a dynamic régime. They are markedly affected by fluid velocity but have relatively low temperature coefficients. A temperature rise of 10° C. at near-ambient temperatures usually increases the rate of a physical process by less than 50%. A chemical régime, on the other hand, is characterised by little or no dependence on the rate of stirring and high temperature coefficient. A rise of 10° C. may double or treble the reaction rate. If a particular chemical reaction should exhibit the opposite characteristics of a low temperature coefficient and high dependence on rate of stirring, it is likely to scale up in accordance with the rules for a dynamic régime.

The dynamic régime in fluid systems is divided into gravity-controlled, viscosity-controlled, and surface tension-controlled. The thermal régime is divided into conduction, convection, and radiation-controlled. The chemical régime is divided into homogeneous and heterogeneous. The heterogeneous class divides further into systems having a fixed (fluid-solid) interface and those in which the interface is mobile (fluid-fluid).

Sometimes there are two or more rate-determining processes, each of which scales up according to a different law. This constitutes what is called a Mixed Régime. A familiar example is the generalised fluid friction equation in which the pressure coefficient is shown as a function of the Reynolds and Froude numbers (equation (2). To scale up according to the Reynolds number the fluid velocity requires to vary inversely as the linear dimension, whilst according to the Froude number it should vary directly as the square root of the linear dimension. No scale-up procedure is possible unless the effect of one of these dimensionless groups is either calculable or can be rendered negligible. In closed and completely-filled liquid systems the effect of the Froude group is negligible and equation (2) simplifies to equation (3). In tank tests of model ships' hulls the Froude number is the dominating factor and the effect of the Reynolds number is calculated and allowed for.

The Future

What is needed today in the field of model theory applied to chemical engineering is a great deal more data on the actual performance of small- and large-scale equipment under corresponding conditions, so that a better appreciation can be reached of the degree of accuracy to be expected from the application of scale-up principles and rules. It would, for example, be a useful exercise for students to construct a model or model element of a piece of operating plant, test it under appropriate conditions and compare the predicted and actual performance of the prototype.

A second requirement is for some improvement on the empirical power function relationship between dimensionless groups. The customary empirical exponents vary quite considerably according to conditions ; for example, in equations for heat transfer by forced convection the exponent on the Reynolds number may vary from 0·5 to 0·8 according to the geometry of the system. A less empirical form of function would allow of more accurate and reliable extrapolation.

Improvements along either of the above lines would enable more information to be obtained in less time from smaller and cheaper models or pilot plants. As our theoretical knowledge of chemical engineering increases it is possible that the pilot-plant stage in process development may come to be omitted more often; yet ultimately it is a question of balancing the costs of pilot scale development against higher capital costs and more prolonged teething troubles on the main plant when the pilot stage is by-passed. The greater the economies that can be made in small-scale experimentation by the application of model theory, the more frequently will it be advantageous to build and test a pilot plant. In any case, whether the construction of a pilot plant is envisaged or not, a theoretical scale-up study of a new process can often enable subsequent operating difficulties to be avoided.

Symbols Used

d = diameter of mixer paddle.
g = acceleration of gravity.
L = linear dimension.
m = exponent on modified Reynolds number in mixing.
N = rotational speed of mixer paddle.
P = power consumption.
p = pressure.
t = time.
u = linear velocity parallel to x co-ordinate.
v = linear velocity parallel to y co-ordinate.
w = linear velocity parallel to z co-ordinate.
x = exponent on Reynolds number in fluid friction.
α_x = angle between the x co-ordinate and the vertical.
Δ = increment (*e.g.* Δp, increment in pressure).
ϕ = function (unspecified).
μ = viscosity.
ρ = density.

These variables may be expressed in any consistent units in which force and mass are not defined independently (if they are, the Newton's Law Constant g_c will appear in certain dimensionless groups).

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THE SCALING-UP OF REACTORS FROM A KNOWLEDGE OF KINETICS

By Prof. K. G. DENBIGH, D.Sc., M.I.Chem.E.*

SUMMARY

The paper is concerned with the kinetic factors which determine whether the best yield of a reaction may be obtained by batchwise operation, or by use of a tubular reactor, or by a set of continuously stirred tank reactors. In complex reactions, such as are frequently met in the production of organic chemicals, one or other of these three types of operation will usually give the highest yield, and this may sometimes be still further improved by the use of a controlled sequence of temperatures. Thus whenever the yield is one of the important economic factors, the process should be "tailor-made" to the particular type of reaction kinetics. This is a necessary part of the scaling-up procedure.

Introduction

In the design of any large-scale reaction process on the basis of laboratory work, there are commonly two important decisions to be taken which precede any detailed design of the equipment:

(1) whether the process should be operated batchwise or continuously and, if the latter, whether a tubular reactor or a set of stirred tank reactors, should be used, and

(2) what should be the necessary volume of the chosen type of reactor for the required output.

The calculations needed for (2) above are already well known and will be referred to only very briefly. The bulk of the paper will be concerned with the most appropriate choice of the type of process—batch or continuous—in so far as it is affected by the kinetic factors which determine the yield of the particular reaction. The question to be discussed is thus: Which type of process gives the largest yield of the required product? All other economic aspects, such as labour usage, controllability, etc., will be left aside.

The term "yield" is to be understood here in the same sense as in organic chemistry (and not as output per unit of time and volume). For a reaction A + B = C, an 80% yield means that 0·8 moles of C are obtained for every mole of A or B (whichever is not in excess), the remaining 20% being accounted for either as incomplete conversion of A and B, or as unwanted by-products due to side reactions or consecutive reactions. In the production of organic chemicals the yield is often of decisive importance in determining whether or not a process is competitive, and the point to be made is that the yield may be appreciably improved by an appropriate choice of the type of process, and also by the sequence of concentration changes and temperatures.

Batch and tubular reactors

Consider first the relatively small difference between a batch reaction, as used in laboratory work, and continuous reaction in a tubular system. In any element of reacting fluid passing through a tubular reactor there is a continuous change of composition, as in a batch process. However, the fluid elements at different distances from the axis have different times of passage and there also occurs molecular or eddy diffusion from one element to another. For this reason not all molecules have the same time of passage; fluid moving near the axis may not react sufficiently and fluid moving near the wall may react for too long a time, especially if there are degradation reactions. This applies particularly if there is laminar flow. (For example in a polymerisation process, using highly viscous liquids, the distribution of residence times would tend to broaden the molecular weight distribution of the polymer, as compared to the same reaction carried out batchwise.)[1] On the other hand, under turbulent flow conditions the distribution of the residence times is not very large and the assumption of "plug flow" becomes a fairly good approximation. Under such conditions the tubular reaction process is almost equivalent in its chemical kinetics to the corresponding batch process whose duration is equal to the time of passage of the " plug " and which has the same feed composition and distribution of temperatures.

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Continuously stirred tank reactor

A very different situation prevails in the continuously stirred tank reactor (C.S.T.R.). If mixing is almost perfect, as is fairly easily attained in practice, the outflow composition is the same as the composition within the vessel. The stationary concentrations of reagents are therefore much lower than the corresponding average concentrations in the batch or tubular reaction system.

For example, in the latter types of process, if equal volumes of solutions of the reagents A and B are mixed together, each of 10 units concentration, then the concentration of each reagent at the moment when reaction begins is therefore 5 units and thereafter it falls steadily (Fig. 1). Let the duration of the process be such that the final concentrations are 0·5 units, *i.e.* 90% conversion. If a continuously stirred tank operates on the same feed solutions, and is large enough to give the same degree of conversion, the outflow concentrations must be 0·5 units and these values therefore prevail everywhere in the vessel, on the supposition of perfect mixing. Thus, whereas the batch or tubular reaction processes operate with concentrations which fall steadily from 5 units to 0·5 units, the C.S.T.R. operates all the time at the latter concentration (Point Q on Fig. 1).

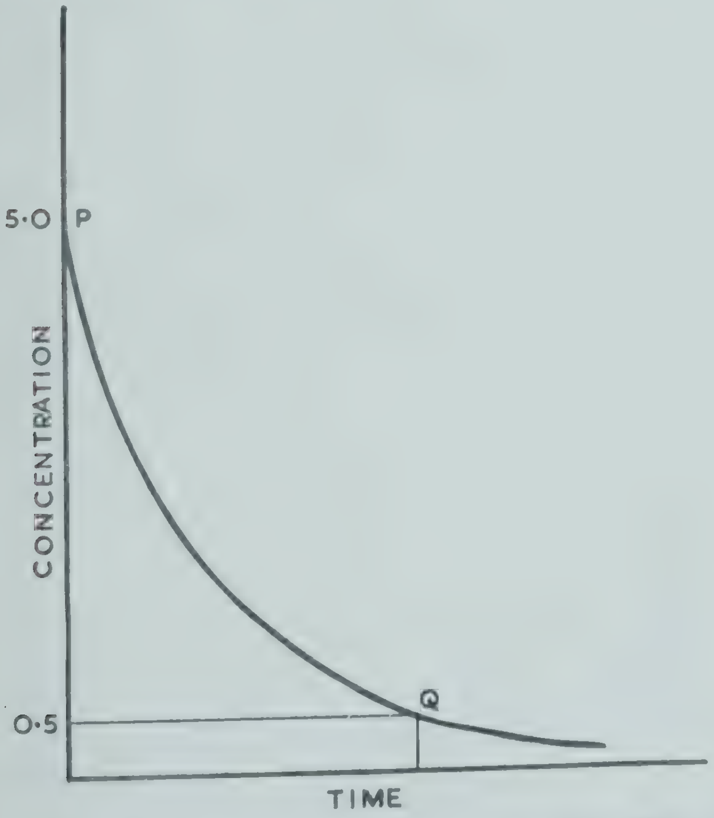

Fig. 1.—*Progressive and stepwise concentration changes.*

The entering solutions of A and B are mixed into this reacted liquor as soon as they are added and never pass through a stage of relatively high concentrations.

The average concentration of the reagents and the average reaction rate, is therefore much lower in the C.S.T.R. than in the corresponding batch or tubular reaction processes for the same

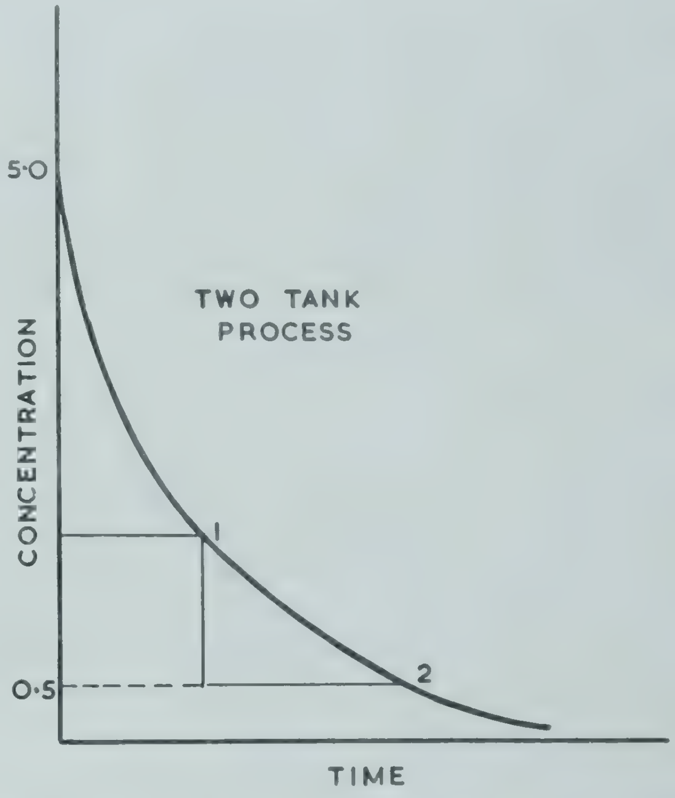

Fig. 2A.—*Stepwise concentration changes for 2-stage C.S.T.R.*

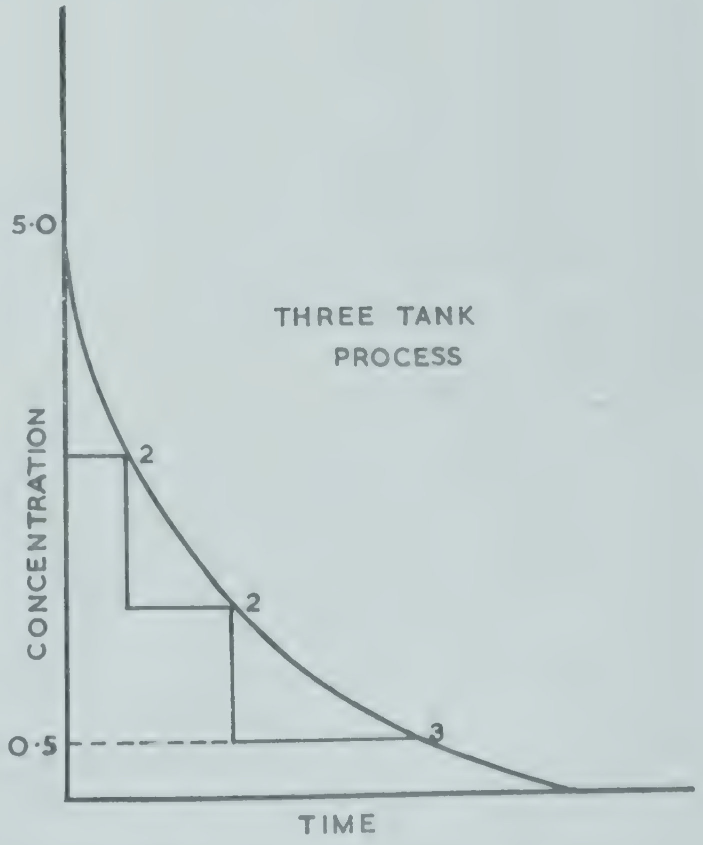

Fig. 2B.—*Stepwise concentration changes for 3-stage C.S.T.R.*

feed conditions and degree of conversion. This conclusion has two important consequences:

(1) the volume of the C.S.T.R. must be much larger than required for a batch or tubular reactor for the same rate of output; to reduce the difference it is customary to use two or more tanks in series, resulting in a stepwise fall of concentration (Fig. 2), and

(2) the yield of the desired product (which is quite distinct from the degree of conversion of the reagents, if there are side reactions or consecutive reactions), may be either greater or less than in a batch or tubular reaction process. This effect will now be discussed in relation to a selection of the various kinetic factors which may affect the yield.

Effects on Yield

Effect of a product of reaction

Consider as an example an organic nitration reaction, in which the yield of the required nitro-body is often very sensitive to the concentration of the nitrating acid, because the speed of side reactions relative to the main reaction is affected by the acid concentration. Moreover one of the products of reaction is water.

$$RH + HNO_3 = RNO_2 + H_2O.$$

This water serves to dilute the acid as reaction proceeds. It follows that the *instantaneous yield*, which is to say the yield from each infinitesimal amount of RH which is nitrated, varies during the course of the process.*

Two important cases arise in practice. The first, illustrated in Fig. 3, is where the instantaneous yield falls continuously with increasing dilution of the acid. In a batch or tubular reaction process, if P and S are the initial and final acid concentrations respectively (the value of S being determined by the amount of excess acid which is used), the overall yield is a weighted mean of the instantaneous yields over the reaction trajectory PQRS. On the other hand, using three C.S.T.R. in sequence, if the feed acid to the first tank is chosen as P, as in the batch process, the stationary acid concentration in the first tank will not be P but will be at some lower value such as Q, for the reasons discussed. In the subsequent two stages the acid concentration will have values such as R and S. The overall yield will therefore be the weighted mean of the yields obtained at the points Q, R, and S—weighted according to the relative amounts of reaction in the three tanks. It is evident that, through the lower average acid strength, this type of process inevitably gives a lower yield than is obtainable from the batch or tubular reaction processes. The best that can be done with the C.S.T.R. in this situation is either (*a*) to use a larger excess of nitrating acid (but this may be uneconomic), (*b*) to increase the number of tanks in sequence, or (*c*) in certain instances to divide the total feed of the organic compound RH between the first and second tanks. If sufficient quantitative information on the yield characteristics is available, it may be

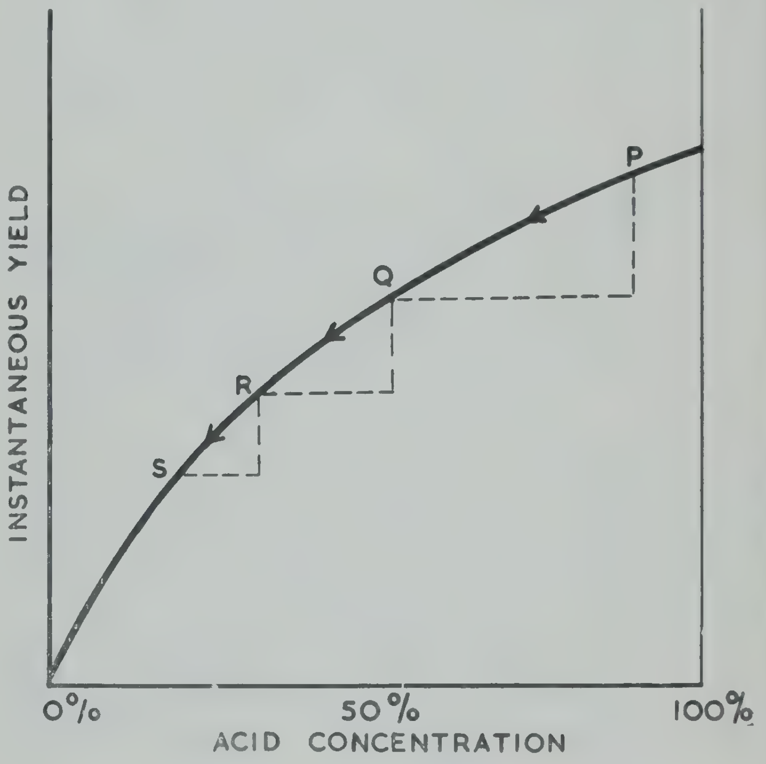

Fig. 3.—*Instantaneous yield curve without a maximum.*

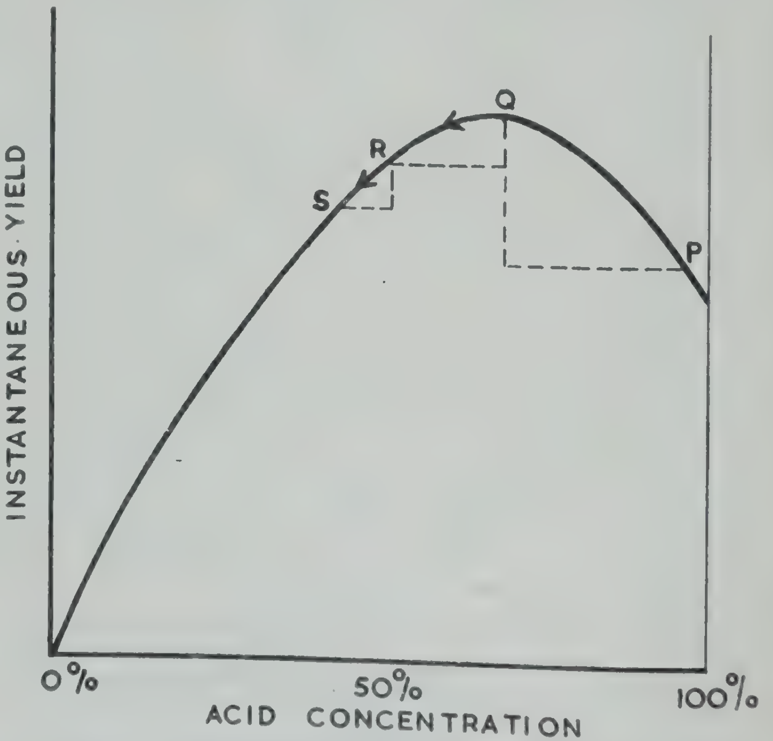

Fig. 4.—*Instantaneous yield curve with a maximum.*

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* The concept of instantaneous yield is further elucidated in connection with equations (4) and (5) below.

possible to decide which of these procedures is preferable, and also to select the best relative sizes of the tanks.

In the second case, illustrated in Fig. 4, the yield is at a maximum at a particular concentration of the acid, and it is evident that the C.S.T.R., if correctly designed, may give a higher yield than can be obtained from batchwise or tubular reactors. As in the first instance, the latter types of process give instantaneous yields over the whole of the trajectory PQRS. By contrast, for the same feed acid P, the optimum acid concentration Q can be maintained as a stationary state in the first tank of a C.S.T.R. system ; if this tank is made sufficiently large, relative to the succeeding ones, the greater part of the overall conversion can be made to occur at the point Q, where the instantaneous yield is at its highest value.

*Effect of the order of reaction**

Consider two competing reactions

$$2\text{A} + \text{B} = \text{X}$$

$$\text{A} + \text{B} = \text{Y}$$

in which X is the desired product and Y is a wasteful by-product. It may be that the two reactions are not of the same kinetic order. Suppose that the reagent B is in large excess and that the rate of the X-forming reaction varies as the square of the concentration of A and the rate of the Y-forming reaction varies as its first power. Thus

$$\frac{dx}{dt} = k_1 a^2 \quad . \quad . \quad . \quad (1)$$

$$\frac{dy}{dt} = k_2 a \quad . \quad . \quad . \quad (2)$$

where a is the momentary concentration of A and dx and dy are the numbers of moles of X and Y respectively which are formed in unit volume in a time dt.

Hence

$$\frac{dx}{dy} = \frac{k_1 a}{k_2} \quad . \quad . \quad . \quad (3)$$

The ratio of the amounts of X and Y formed during the interval dt, and therefore the yield of X, is evidently greater at higher concentrations of A, and will diminish during the course of the reaction.

In general, when the useful reaction is of higher order than the wasteful reaction, good yield is favoured by raising the concentration of the reagent. Conversely if the useful reaction is of lower order, the yield is favoured by lowering the concentration. (This reduces the rate of reaction and therefore requires the use of a larger reactor. The discussion is based on the supposition that it is yield rather than rate which is economically important.)

In the former case it is evident that the batch or tubular reaction processes are to be preferred, since they operate at higher average concentrations, for the same feed conditions. If, for quite different reasons, there are grounds for choosing the C.S.T.R., the yield can be raised—though not as high as the batch yield—by increasing the number of tanks in sequence.

Where the useful reaction is of the lower order, the concentration of the reagent should be as low as possible. If, however, it is undesirable to reduce the concentration of the feed solution, the C.S.T.R. system would give a higher yield than the batch or tubular reaction systems, and especially if the first tank in the sequence were large, to provide a low concentration of A immediately on mixing. Alternatively much the same effect could be obtained by operating a type of batch or tubular reaction process in which successive small increments of the reagent A were added to the mass of reagent B, at a rate small compared to the rate of reaction, so that the concentration of A was always at a low level.

The above example of two competing reactions may be used to illustrate the distinction between instantaneous and overall yields. In a batch process, during the interval dt, dx moles of X are formed and $-da$ ($= 2dx + dy$) moles of A are used up, per unit volume. Thus the instantaneous yield of the useful product X is

$$\phi = -2dx/da \quad . \quad . \quad (4)$$

where the factor 2 arises from the stoichiometry of the reaction. Let a_0 and x_f be the initial number of moles of A and the final number of moles of X respectively, per unit volume. Then the overall yield* is

$$\Phi = 2x_f/a_0 \quad . \quad . \quad (5)$$

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* The following is based on one of the author's previous papers.[2]

* An alternative definition which is more suitable when any unchanged A is recoverable is

$$\Phi = 2x_f/(a_0 - a_f),$$

where a_f is the final concentration of A.

For a batch process, where a, x, and y vary continuously, equation (4) can be integrated between the initial and final states of the system :

$$x_f = -\tfrac{1}{2}\int_{a_0}^{a} \phi \, da \qquad . \qquad . \qquad (6)$$

Substituting equation (6) in equation (5), the overall yield is obtained as an integral over the instantaneous yields

$$\Phi = -\frac{1}{a_0}\int_{a_0}^{a_f} \phi \, da \qquad . \qquad . \qquad (7)$$

Similarly, in the case of a C.S.T.R., the overall yield may be expressed as a summation (rather than as an integral) of the yields characteristic of each tank in the sequence.

These integrals or summations may be evaluated in an explicit form whenever the kinetics of the competing reactions are known. For instance in the example discussed above, using the kinetic equations (1) and (2), we obtain

$$\begin{aligned} \phi &= 2dx/(2dx + dy) \\ &= 2k_1a^2/(2k_1a^2 + k_2a) \qquad . \qquad (8) \end{aligned}$$

Substituting this result in equation (7)

$$\Phi = -\frac{1}{a_0}\int_{a_0}^{a_f} \left(\frac{2k_1a}{2k_1a + k_2}\right) . \, da \qquad . \qquad (9)$$

This integral is readily worked out in terms of a_0 and a_f, the initial and final concentrations respectively. The term a_f may then be expressed in terms of t, the duration of the batch reaction, by integration of

$$-\,da/dt = 2k_1a^2 + k_2a$$

thus obtaining Φ as a function of a_0 and t :

$$\Phi = 1 - \frac{r}{b} + \frac{r}{a_0}(k_2t + \ln r/b) \qquad . \qquad (10)$$

where

$$r = k_2/2k_1$$

and

$$b = (a_0 + r)\exp(k_2t) - a_0.$$

Degradation Reactions

Another example of the possibility of predicting the best type of process occurs in degradation reactions. This name will be given to those reactions in which the required product X can react further to give some unwanted product Y. This occurs in consecutive reactions :

$$\begin{aligned} \mathrm{A} + \mathrm{B} &\rightarrow \mathrm{X} \\ \mathrm{X} + \mathrm{B} &\rightarrow \mathrm{Y} \end{aligned}$$

or in reactions of the type :

$$\mathrm{A} + \mathrm{B} \begin{array}{l} \rightleftharpoons \mathrm{X} \\ \rightarrow \mathrm{Y}, \end{array}$$

where the X-forming reaction is reversible and the Y-forming reaction is relatively slow. An example of the first is the halogenation of an organic compound, the mono-halogen derivative being the required product, whilst higher derivatives are unwanted.* Another important example is the oxidation of ammonia : nitric oxide is the required product, but nitrogen is mainly obtained if the time of contact with the catalyst is too long. Similarly in the oxidation of methanol to produce formaldehyde, where carbon dioxide may be formed by degradation.

In both the above types of reaction scheme the concentration of the required product X passes through a maximum at a certain time t_m. Therefore, if it is desired to obtain the maximum yield of X, relative to the amount of A and B introduced into the system, the reaction process should have a duration t_m, no shorter and no longer.

It can be seen that the yield of X has a higher maximum for a batchwise reaction than for the same process carried out in a tubular reactor or a C.S.T.R. In the batch, the entire contents of the reaction vessel can be given a time of reaction equal to t_m. In either type of continuous process, on the other hand, there is a distribution of residence times ; even though the mean time of passage is given its optimum value, a considerable fraction of the molecules will have residence times both appreciably shorter and appreciably longer than the optimum. In brief, the probability that a given molecule of A becomes transformed into X (and not into Y) has a maximum at a certain time ; therefore any spread in the residence times will reduce the maximum yield.

An ideal plug-flow reactor would theoretically produce results identical with a batch reactor, as would an infinite (effectively infinite) cascade of C.S.T.R.'s; but any practical reactor (which cannot do more than approximate to plug flow) would show this spread of residence times to a greater or less extent.

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* For an interesting study of the chlorination of benzene see MacMullin.[3]

To examine the magnitude of the effect, it is convenient to discuss the two extreme cases, batch reaction and a single C.S.T.R., for which the spread of residence times are least and greatest respectively. Following MacMullin[3] the reaction sequence

$$\mathrm{A} + \mathrm{B} \rightarrow \mathrm{X}$$
$$\mathrm{X} + \mathrm{B} \rightarrow \mathrm{Y}$$

will be discussed and it will be supposed, for simplicity, that the rate of the first reaction is proportional to the concentration of A and the rate of the second reaction is proportional to the concentration of X, as might be the case if B were in large excess. Thus in a batch reaction

$$-\,da/dt = k_1 a \quad . \quad . \quad . \quad (11)$$

$$dx/dt = k_1 a - k_2 x \quad . \quad . \quad (12)$$

where a and x denote concentrations of A and X respectively.

Solving these equations we obtain

$$x = \frac{a_0}{(r-1)}\,(\mathrm{e}^{-k_1 t} - \mathrm{e}^{-k_2 t}) \quad . \quad (13)$$

where a_0 is the initial concentration of A and $r = k_2/k_1$. The maximum value, x_m, is obtained at a time t_m given by

$$t_m = \frac{1}{k_1(r-1)} \ln r \quad . \quad . \quad (14)$$

which when substituted into equation (13) gives

$$x_m = a_0 \exp\left(\frac{r}{1-r}\,.\,\ln r\right) \quad . \quad (15)$$

Thus the maximum yield which may be obtained by a batch process, when computed relative to the amount of A introduced into the system, is

$$\Phi_m = \exp\left(\frac{r}{1-r}\,.\,\ln r\right) \quad . \quad (16)$$

For the same reaction process carried out in a single C.S.T.R., the equations corresponding to equations (11) and (12) are

$$u\,(x + y)/V = k_1 a \quad . \quad . \quad (17)$$

$$ux/V = k_1 a - k_2 x \quad . \quad . \quad (18)$$

where a, x, and y are the volume concentrations of A, X, and Y respectively in the outflowing liquid, u is the volume rate of flow and V is the volume of liquid in the vessel. The yield of X is

$$\Phi = x/(a + x + y) \quad . \quad . \quad (19)$$

and substituting from equations (17) and (18) this becomes

$$\Phi = k_1\bar{t}/(1 + k_1\bar{t})(1 + k_2\bar{t}) \quad . \quad . \quad (20)$$

where $\bar{t} = V/u$, the mean time of passage. This yield has a maximum value at a time $\bar{t}_m$ given by

$$\bar{t}_m = (k_1 k_2)^{-\frac{1}{2}} \quad . \quad . \quad (21)$$

Substituting in equation (20), the maximum value of Φ for the C.S.T.R. is

$$\Phi_m = (1 + r + 2r^{\frac{1}{2}})^{-1}. \quad . \quad (22)$$

The extent to which the maximum batch yield, as given by equation (16), is greater than the maximum yield obtainable from the single C.S.T.R. thus depends on the ratio, r, of the velocity constants. The effect is greatest when the velocity constants are equal, as is probably approximately the case in many reactions involving successive substitution. Under these conditions a solution of equations (16) and (22) shows that the batch yield is 47% larger, which is considerable.

The theory could be elaborated by allowing a monetary value to the product Y, as well as to X and in this case the advantage of batch operation would be reduced. For three successive reactions

$$\mathrm{A} + \mathrm{B} \rightarrow \mathrm{X} \rightarrow \mathrm{Y} \rightarrow \mathrm{Z},$$

the reader is referred to MacMullin's paper[3] and also to Corrigan and Young.[4]

Optimum Temperature Profiles

So far the yield has been discussed in terms of the influence of composition and the spread of residence times. It has perhaps not been sufficiently realised that the yield of a complex reaction may often be appreciably improved by making a continuous change of temperature during the course of the reaction.[1] For this purpose the tubular reactor is in some respects the most convenient, although a controlled change of temperature can also be applied during the course of a batch process. In a C.S.T.R. of several tanks in sequence, each tank can be held at a fixed temperature, with stepwise change from one to the next, but this is less effective in principle than a continuous variation.

Consider the very general formulation of a complex organic reaction shown in Fig. 5 where X is a reaction intermediate, Y is the required products, and P and Q are wasteful by-products due to side reactions. The aim must evidently be to obtain maximum speed of reactions 1 and 3 relative to reactions 2 and 4. Ideally this would be achieved by use of selective catalysts, but a controlled variation of temperature is often helpful, or in some instances as in T.N.T. production a controlled variation of the concentration of the reacting fluid.

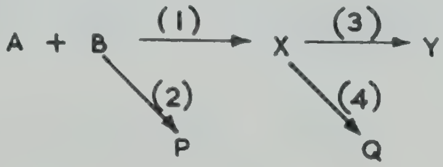

Fig. 5.

Suppose that the temperature coefficient of reaction 1 is less than that of reaction 2, whilst the temperature coefficient of reaction 3 is greater than that of 4. It would evidently be advantageous to use a low temperature in the early stages of the reaction process, whilst X is accumulating, and a much higher temperature in the later stages when the predominant reaction is the conversion of X into Y. This choice of the temperature history will favour the speed of reaction 1 relative to reaction 2 and of reaction 3 relative to reaction 4.

If the temperature coefficients of reactions 1 and 4 are greater than those of reactions 2 and 3, there would be an advantage in using a falling temperature along the path of the reaction.

In each instance therefore it may be expected that there exists a certain temperature profile which will tend to give the optimum yield of the desired product. In any actual instance the information on the magnitude of the temperature coefficients, or even on the existence of the transient intermediates, may not be available, and in any case the mathematical theory of the shape of the profile has not yet been established. But practical experience in explosives production has shown that to find the optimum sequence of temperatures by trial and error can sometimes be extremely effective in improving the yield.[5]

Complex reactions of the above type are not the only instances which give rise to an optimum temperature profile. The example of reversible exothermic reactions (*e.g.* ammonia synthesis, the water gas reaction, sulphur dioxide oxidation) is well known.[1, 6, 7] Another important class is that of the degradation reactions, as discussed recently by Bilous and Amundson.[8]

Calculation of the Capacity of the Reactor

When the type of process has been decided, together with the choice of the inlet concentrations, the next step in the process design is the calculation of the capacity of the reactor which is necessary for a given output and yield. This is bound up with the problem of heat transfer. However, in many instances it will be possible first of all to calculate the necessary capacity of the reactor for some desired temperature, or temperature sequence, and subsequently to determine the heat transfer conditions which are needed to achieve this.

The first part of this problem is one of reaction kinetics. For a batch reaction it is a question simply of integrating the familiar differential equations, although, if the temperature is not constant, a graphical integration will probably be needed. Similar considerations apply to the tubular reactor, except that it may be necessary to correct for deviations from plug flow, for example by determining the distribution of residence times and using this instead of a simple time interval in a graphical integration. When a C.S.T.R. is used, the basic equations for the calculation of the reactor capacity are algebraic rather than differential and are of the type of equations (17) and (18) above. These take on an iterative character when there is more than one tank in the sequence, as is normally the case.

The interested reader may find it useful to consult the first reference listed below, which refers to the work of MacMullin and Weber, Kirillov, Bosworth, Piret, and others.

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THE ECONOMIC ASPECTS OF SCALING-UP CHEMICAL PLANTS

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SUMMARY

After a comment on the desirability of economy in chemical plants the principles of "Economies of Increased Dimensions" are stated and developed, and attention is drawn to the limitations in practice. Examples are given of the correlation of cost to size for individual units with some of the underlying explanations. The effect of scaling-up in size on the efficiency of units and the operating manpower requirements are discussed briefly. A detailed example of the effect of scaling-up a complete plant is given in estimates of capital and production costs for a plant at three different scales of output.

The paper concludes with a critical consideration of some previously published information on factors which can be used in scaling estimates of construction costs of chemical plants.

Introduction

When problems of scaling-up and size of chemical plant are being considered, the engineer will find there are many decisions that have to be made which cannot properly be decided solely on technical grounds. The question arises whether any economic advantage can be gained in scaling-up the size of individual units and the effect of such a course upon the economics of the plant as a whole.

In practice the engineer is usually influenced by his experience that scaling-up does provide economies, and he will be well aware of the desirability of this as well as the need to ensure that the design is technically sound. However, there may be a tendency for the practical engineering problems to take priority and the economic problems left to look after themselves unless certain principles are always borne in mind.

It is not intended to discuss economic theory in this paper, but rather to demonstrate the principles by examples and to give an indication of the extent of the economies which become possible when scaling-up plant.

Principles

The advantages of large-scale production usually reflect changes both in the type and amount of production equipment and in techniques and management methods as compared with production on a smaller scale. A reduction in unit cost is usually effected by the substitution of fixed factors of production for variable factors, *i.e.* machinery and equipment for labour. In general, the chemical manufacturing industry has already gone a very long way in this direction and the trend is still continuing.

The design phase of chemical plants is therefore a very important stage in which the pattern of the costs of production are mainly decided.

When techniques have been considered and process data are available, the selection of equipment follows. The scale of the plant as a whole may have been decided as a result of market surveys and within these terms of reference the engineer makes the choice of the necessary individual units with the aim of achieving the lowest possible total unit cost of the product at, and approaching, the design output.

The principles which will serve as a reminder in the choice of the size of individual units, are simply stated in the "Economies of Increased Dimensions":[1]

(1) A large machine or structure is usually cheaper to construct per unit of capacity or output than a smaller machine or structure.

(2) There is often a purely mechanical advantage (higher efficiency) to be gained by using large machines.

(3) The operating manpower required for a large unit is often no more than that required for a smaller unit.

Practical Limitations to Increasing Size of Units

Engineers will readily be able to find examples within their experience to substantiate these principles. It will probably also be appreciated that there are limitations to their application. There cannot be an infinite extension of economies in practice at any set time.

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The line of demarcation between decreasing and increasing cost per unit of capacity with increasing size of items of chemical plant is not always clear. Usually border-line cases can only be resolved after fairly lengthy investigation with manufacturers of the particular type of equipment. Very often the limiting factor is not that of the extrapolation of design from proven unit sizes, but rather that of the capacity of the capital equipment available for the manufacture, transport, and erection of the plant. Therefore, in the short term it may be almost impossible to obtain very large items of plant although they may appear to be economically advantageous. If, for example, a manufacturer of paper-making machinery were asked to supply a calendering machine bigger than anything he had made before, investigations might prove that the only limit was the size of rubber-covered rolls which were readily obtainable. Consultation with the specialist makers of the rolls might show that they would be prepared to extend their own plant with new cranes, lathes, and grinding machines capable of handling the new sizes if demand were sufficient to justify the investment. They would then be able to supply the larger size required some years after the original request.

The capital expenditure incurred by the roll makers would have to be recovered from the estimated demand in a reasonable period and might result in proportionately higher prices for the orders received during that period.

Demand for larger plant will usually only arise if business organisations have expectations that its use, even at enhanced prices, will increase their profits, but when this is the case the boundaries of demarcation in the economical size of plant readily available for later schemes are pushed upward.

Considerable economies have been achieved by the Central Electricity Authority through adopting ever-increasing sizes of turbo-alternator sets and boilers in power stations. Rapid technical progress has resulted in the installation of sets of 200 M.w. capacity whereas only a few years ago it was considered that 60 M.w. sets were the largest possible size. In this case, examination of the published facts[2, 3] indicates that the economies are not so much due to reduced fixed costs per k.w. of installed capacity, as to the higher efficiency of the new steam cycles adopted (which are only possible with the larger capacity units) and to the reduced labour cost.

These two examples show that, providing demand is sufficient to justify the applications, design and production problems can be overcome in the course of time with the prospect of increasing economies as unit-size increases.

In the short term however, the case may be different. In order to enter a market quickly it may be necessary for the engineer to make his choice from the available sizes of chemical plant since manufacturers require time and, usually, increased payment to develop items in larger sizes even though they are within their manufacturing capacity. Circumstances may arise where standard equipment, although larger than necessary, may cost less than a non-standard article which another manufacturer may make specially to the exact size required.

Comparison of Fixed Capital Costs in Relation to Size

To demonstrate the principle of decreasing capital cost per unit of output as capacity increases in single items of plant, a number of charts are given. In all cases, capital cost to the user is plotted against a measure of output or capacity on a log-log grid. The slope of the line obtained by connecting the point plots, corresponds to the exponent " x " in the following comparison formula.

$$P_2 = P_1 \, (X_2/X_1)^x$$

Where P_1 = price of the first " unit "
P_2 = price of the second " unit "
X_1 = capacity of the first " unit "
X_2 = capacity of the second " unit ".

Data have already been published showing similar correlations of cost to capacity,[4] but unfortunately it has been almost entirely of American origin. Reference to these articles will confirm and amplify the trends shown in the examples given in this paper.

In all cases where prices or values are given in this paper, they are related to levels ruling in November 1956.

Steel pipe, one of the simplest examples, is priced basically on a rate per ton for different ranges of outside diameter. Therefore for pipes of the same thickness, the price within a reasonable range of sizes will vary according to the diameter, whereas of course, the capacity or throughput will vary practically as the square of the diameter. This relationship is shown in Fig. 1 where the slope of the line for 0·375 in. thick pipe is 0·5. However, the slope of the line for the full range of diameters in standard thicknesses, corresponding to equal pressure conditions, is 0·77 although it proceeds in steps.

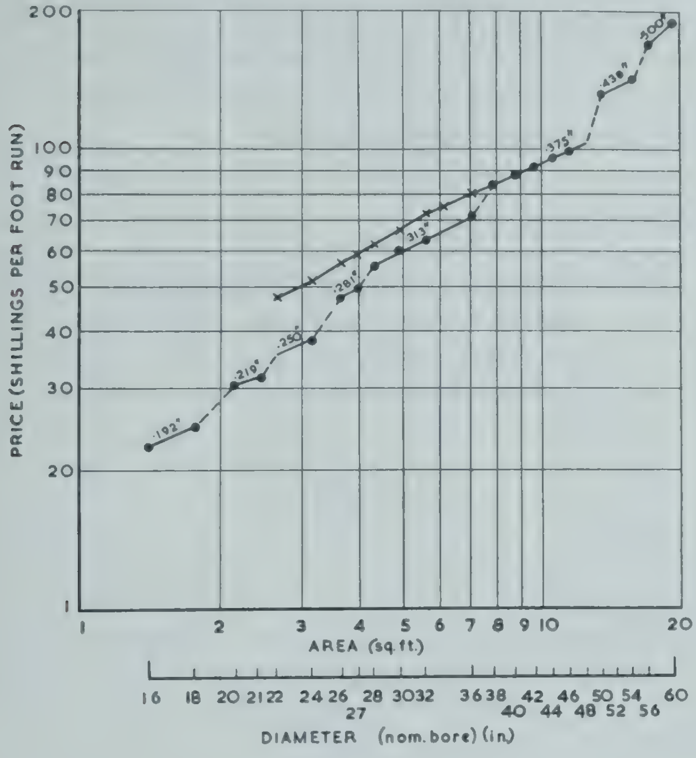


Fig. 1.—*Correlation of cost to capacity for steel pipe. (B.S. 534, Class "A")*

For some equipment there are savings in manufacturing cost per unit of weight as well as savings due to the unit weight decreasing per unit of capacity as size is increased. This may apply over only a small range, and then, as size becomes increasingly larger and specialised expensive equipment becomes necessary, the cost rate per ton may increase considerably. In the engineering industry supplying capital goods it is usual for price quotations to be on the basis of estimated cost with a percentage of profit added. Prices tendered to customers are therefore increased accordingly.

Examples of individual units

STORAGE TANKS (horizontal cylindrical, mild steel)

For the purpose of this example, use is made in Fig. 2 referring to a standard range of tanks which are available for storage of petrol and oil fuel.

Bearing in mind the example of the pipe of different diameters but equal thicknesses, one would expect the price of tanks to vary according to the plate area when using plates of the same thickness. Inspection of the chart will show that over the range of 250 gal. to 2000 gal. this is so, and the exponent in the comparison formula is 0·5. However, it appears that the curve hinges around the point of the 2000 gal. size, and above 2000 gal. to 12 000 gal., the exponent is 0·87. Taking the whole range, an exponent of 0·69 could be used with only a reasonably small error. The exponent of plate area to capacity is almost the same as this value. A number of probable reasons can be mentioned to account for the change of the exponent over the range, such as change in the proportions of cylindrical shell plates to end dishings (the latter being more expensive), the amount of welding to suit stock plate sizes, the size of the equipment, cranes, shop, etc. needed for the larger sizes, and pricing policy which is affected by the numbers of each size which are in demand.

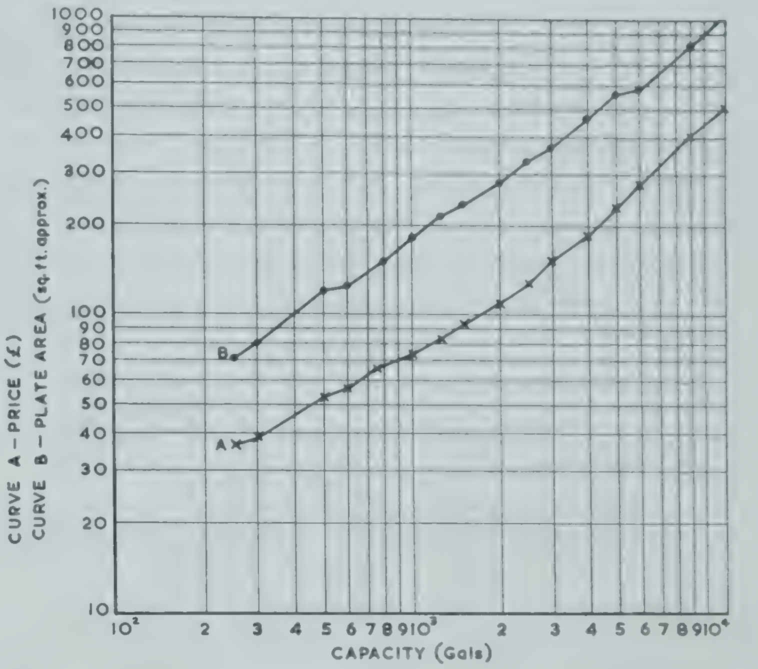


Fig. 2.—*Correlation of cost to capacity for storage tanks (Horizontal Cylindrical, 1/4 in. M.S.)*

The broad issue is clearly demonstrated however —that it is cheaper to use a large tank rather than a number of small tanks of the same combined capacity.

STEAM BOILERS (economic type)

The steam boiler is a specialised form of heat exchanger comprising a pressure vessel with internal tubes held between flat tube-plates. The output of differing sizes will vary in proportion to the heating-surface area available, but the weight and cost of the shell and tube-plates will not, nor will the cost of the boiler fittings.

The variation of price with output for different size units is shown in Fig. 3. Also shown on this chart are the prices of certain auxiliaries which are of interest in themselves. It should be noted that the figures for boiler and auxiliaries do not represent a complete installation, as foundations, lagging, pipework, valves, electrical equipment, cables, etc. are all excluded.

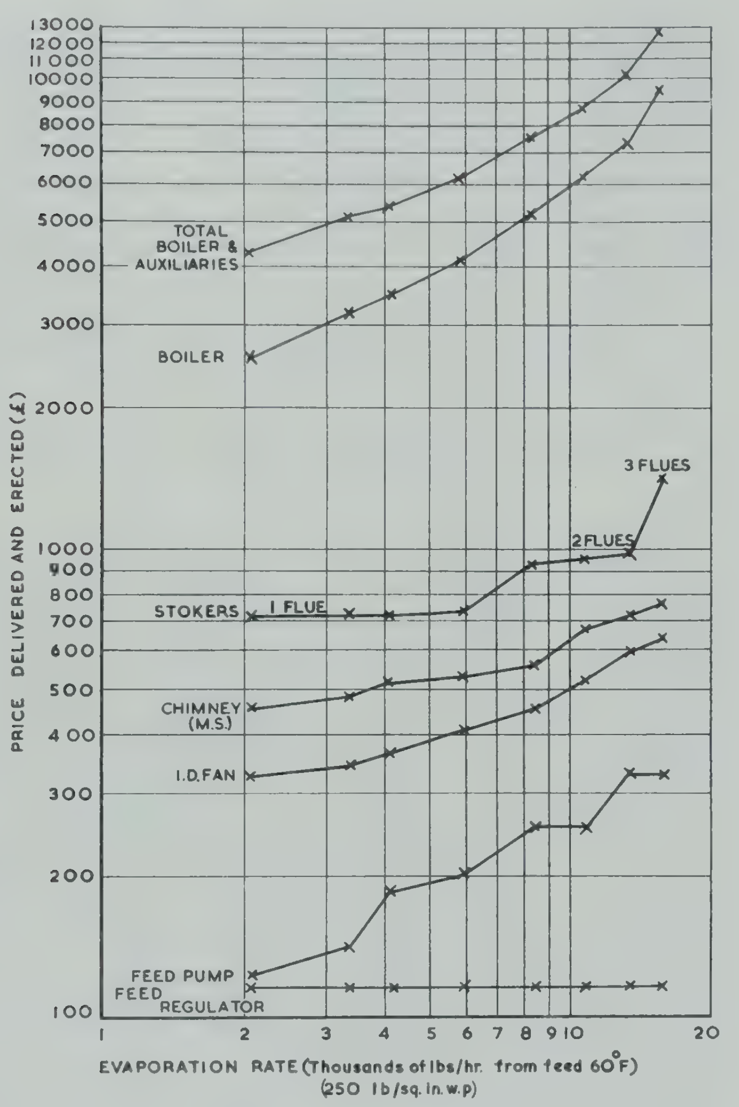


Fig. 3.—*Correlation of cost to capacity for Economic type steam boilers*

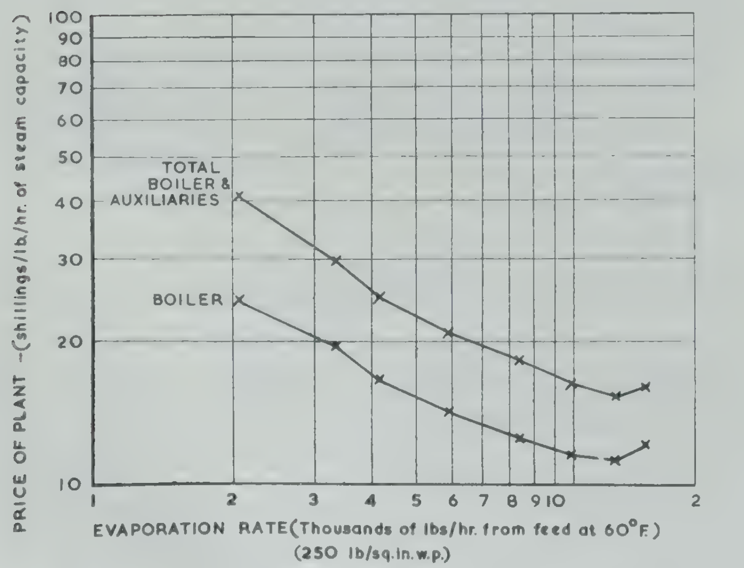


Fig. 4.—*Correlation of cost to capacity for Economic tybe steam boilers*

The average exponent can be taken as 0·655 for the boiler and 0·535 for the boiler with auxiliaries.

Fig. 4 illustrates the unit first-cost per lb. of steam per hour, for the boilers having their total costs given in Fig. 3. The turn up of the curve at the largest size is mainly due to the change in the number of flues required at this point, as it is not practicable to increase the size of the flues any further and it is necessary to have three flues per boiler instead of two.

HEAT EXCHANGER

Fig. 5 shows the variation of price with capacity of a standard range of heat exchangers which are used as after-coolers for air compressors.

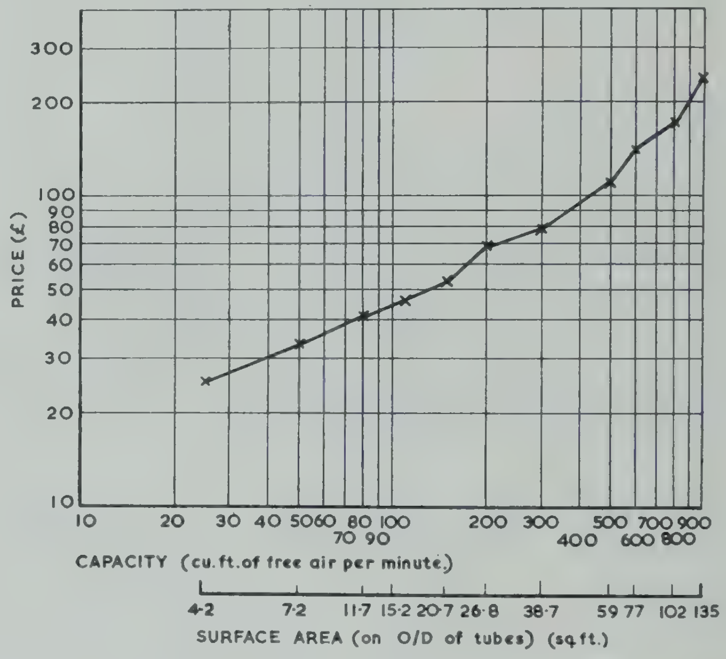


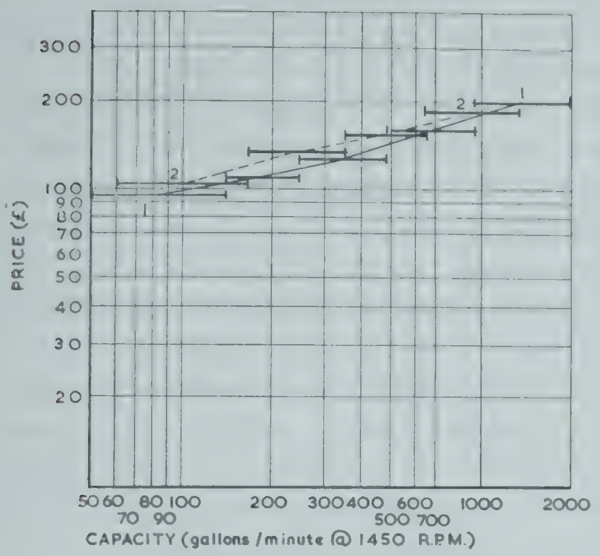
Fig. 5.—*Correlation of cost to capacity for heat exchangers (after coolers)*

Similar principles and slope of curve are observed with other types of heat exchanger.

The exponent for the curve over the whole range can be taken as 0·62.

PUMPS (centrifugal types)

So far the examples have been for static equipment except for the reciprocating pumps and the fans for the boilers which have been regarded as forming part of a unit. Fig. 6 is drawn for a standard range of centrifugal pumps. It is rather difficult to draw curves of a type similar to those previously used when each unit covers a fairly wide field of application with two main variables, such as capacity and head, even when a fixed speed is assumed. For smaller sizes



Curve I for heads between 30 ft. and 60 ft.
Curve 2 for heads between 60 ft. and 90 ft.

Fig. 6.—*Correlation of cost to capacity for centrifugal pumps*

of centrifugal pumps, flow rate and velocity head can be varied widely with only very small changes in size of impeller working within the same basic standard size casing. Thus, fairly widely varying conditions of output can be met with no very significant change in cost. By simplifying the conditions as much as possible, it is possible for curves to be drawn which permit the relationship of output to price to be observed. Within the range plotted the slope for one curve is 0·25 and the other 0·24. It is also interesting to note that the graph for the fan in Fig. 3 has a slope of 0·31 over the range covered.

Increase in Efficiency with Size

Most plant that is thought of as purely chemical plant is static and as such it is not possible to measure its efficiency except perhaps in terms of heat conservation or energy loss due to friction of fluid flow within the static equipment. Unfortunately, too little appears to be known at present to design, say, a heat exchanger on the basis that efficiency of heat-transfer is a function of the relative dimensions of similar units. In practice it appears that whatever calculations are carried out, a good allowance is made for a factor of ignorance and therefore any niceties of calculation which may be possible can, for present purposes, be ignored.

There are, however, many examples of dynamic machinery used in chemical plants or for utilities, where it can be shown that the energy efficiency is greater as the size of the unit is increased. It is in machinery such as prime movers, electric motors, pumps, compressors, etc. that economies in running costs are to be achieved by the installation of larger and more efficient units.

Fig. 7 gives average efficiency curves of pumps and motors and as combined sets. It shows that apart from the variations in efficiency of pumps obtainable from individual manufacturers at each size, there is a well-defined trend towards greater efficiency as the size of the unit increases.

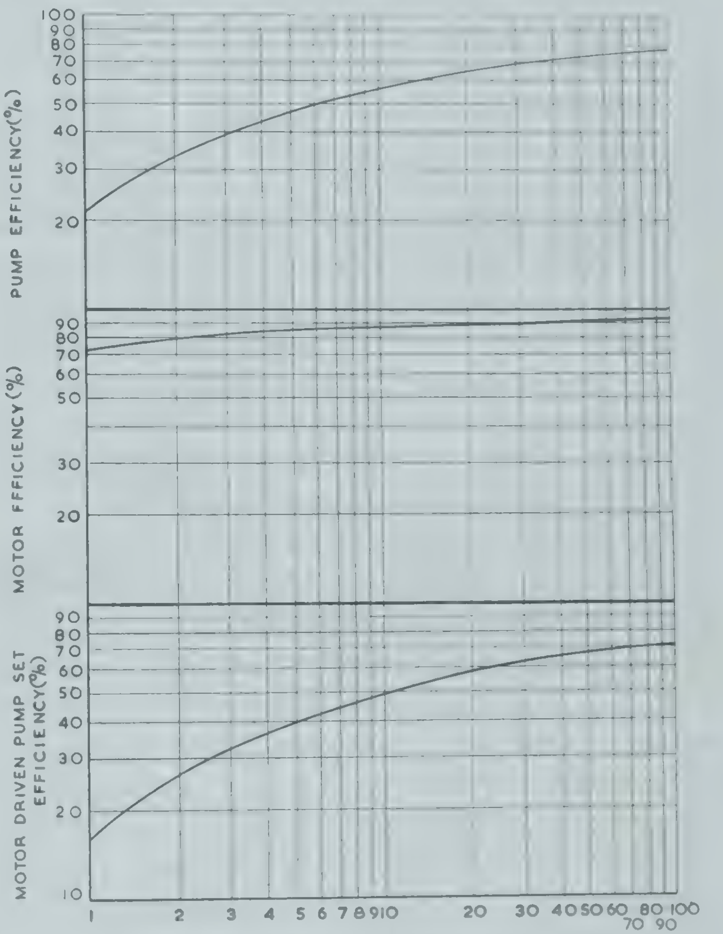


Fig. 7.—*Efficiency of pumps and motors*

Reflection will show that the relationship of efficiency to size is subject to physical laws analogous to these connecting capital cost to size.

Operating Labour

In the same way that one driver is required for a vehicle whether it is a 10 ton lorry or a 5 cwt. van, so a large unit of chemical plant may require no greater labour force than a small unit, although in each case greater skill and responsibility may be needed. If a plant is larger than another due to increased dimensions rather than to multiplication of units, it is possible that the number of operators will not be increased, or if they are, the increase will not need to be in proportion to the output of the plants.

It is probable too that if a larger plant is constructed it may be desirable to incorporate various techniques superior to those which would be employed in a smaller plant. The tendency would be towards labour-saving equipment and instrumentation which would be labour saving.

Application of the Principle in Practice

By his training, the engineer turns first to the determination of the form, dimensions, and materials of the constituent units of the plant to suit the needs of the process. In chemical plant in particular the engineer is guided in his selection by previous experimental data ; commercial-size plants are not the usual place to try out something completely novel which may jeopardise the success of an otherwise promising venture. At this stage the possibility of process obsolescence must be considered—it is wasteful to contemplate installing plant which, by reason of the strength and nature of materials employed, will remain in operating condition for 25 years, if there is sound reason to believe that within say 5 years a more economic process will be developed.

Next, the engineer seeks technical efficiency which by experience leads him to the desirability of large units instead of small. This is conditioned by the need for reliability which may mean that a vital or key item, although within the size or capacity available from one large machine or unit, would be best as two or more units of reduced size so that breakdown of one will allow the plant to run although at reduced capacity. Alternatively, it may be considered that the situation is best met by standby equipment at these crucial points. Load factor also has a considerable influence on the choice ; if a large machine may be running at only a quarter of its normally rated capacity most of the time, and at normal rating only during its peak load, highest efficiency is best obtained by increasing the number of units so that the varying load can be met by running the number of units required while each is operating at or near its maximum efficiency.

If, on the other hand, at the time of installing the plant there is good prospect of expansion in the near future, the engineer will probably be justified in choosing most of the units in the first stage of construction so they are of the largest possible size and thereby economising in first cost and obtaining higher efficiency eventually. In fact, in such circumstances it may be desirable, to install plant larger than necessary to meet immediate requirements, in order to take maximum advantage of the " Economies of Increased Dimensions ". Much careful thought is necessary before making such a decision, otherwise it may happen that the extra depreciation and interest charges on the additional capital spent in anticipation of future output, may more than offset the future savings.

There are many advantages to be gained by the use of manufacturers' standard equipment. This applies particularly to machinery when well-proven designs ensure reliability at reasonable first cost and ease in obtaining spares and replacements. What is often most important is that the equipment can be obtained quickly—sometimes in a fraction of the time required for a special design. The users' choice has to be made from a range of sizes and he normally takes the largest possible size consistent with the foregoing considerations. The advantages of the standardisation of certain sizes within an organisation may over-ride the desirable choice of the largest unit for each application.

Often other considerations, such as safety, may have their effect upon choice of size of equipment, especially if there is a risk of explosive reactions. In such cases units are often kept small and dispersed.

Summarising, it can be said that the good engineer recognises the advantage of choosing plant so that the constituent units have the largest possible size or capacity. By doing this, economies are made in capital charges and in the operating costs. Other considerations have to be taken into account, however, and sometimes render it impossible to take advantage of the utmost economies of size. A very nice balance of judgment is often necessary in order to obtain the wisest outlay of capital in fixed assets.

Comparison of Complete Plants

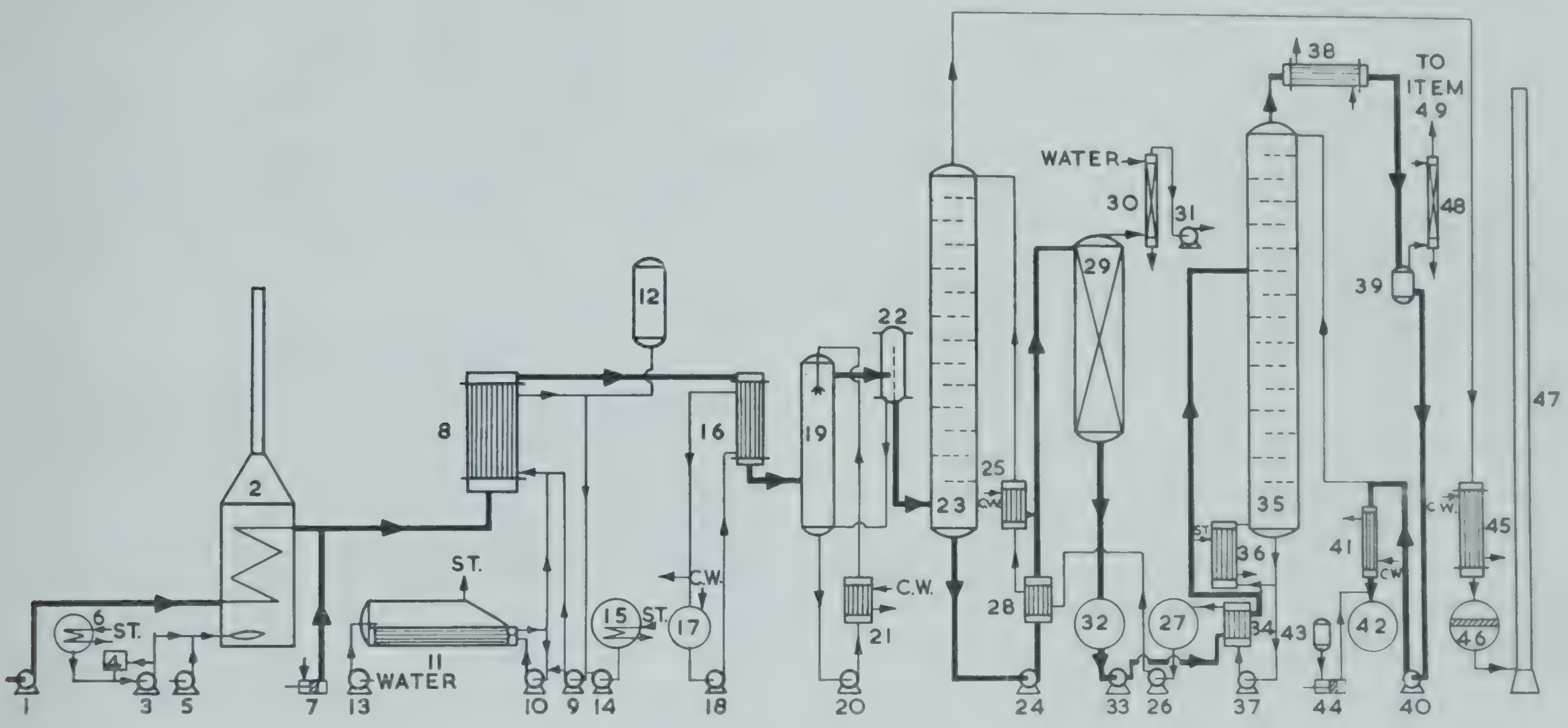
To illustrate the nature and extent of the economies possible in complete plants, an example is given in which the construction cost and unit operating costs of a plant are estimated and compared for three different outputs.

The estimates can be classed as " pre-design " estimates of a fairly high standard. Normally there would be a contingency added to each of the estimates, but as it would probably be a uniform percentage, the comparison of the schemes is not affected and it is accordingly omitted.

The sizing of equipment and the basic information has been provided by the Process Development Division of the Research and Development Department of The Distillers Company Limited, and it has been chosen as an example mainly because it illustrates a fairly broad range of problems and is typical of many catalytic vapour phase processes which are in operation. The sketch flow sheet (Fig. 8) with abbreviated Item List, and the Summary of the Estimate of Construction Cost (Table I) (excluding design costs and consultants' fees, etc.) are supplemented by a chart (Fig. 9) on which the anticipated costs are plotted in a manner similar to the previous examples of individual units.

Background

The economic comparisons were prepared when a pilot plant had already been constructed and



See appendix I for key

Fig. 8.—*Catalytic vapour phase plant. Flow diagram*

Table 1.—*Estimate of Construction Cost for Catalytic Vapour Phase Plant*

Description	Plant A (£)	Plant B (£)	Plant C (£)
Summary of detail sheets :—			
Pump sets with motors and starters	5 675	12 065	27 475
Tanks and vessels, process	1 390	4 480	15 765
finished products	960	4 600	17 440
Columns	7 425	27 550	86 900
Compressors and vacuum pumps	12 210	41 970	58 025
Heat exchangers	6 175	21 225	77 150
General equipment—furnaces, reactors, chimney, gas holders, etc.	14 400	57 900	205 550
Total	48 235	169 790	488 305
Erection of plant (other than that assembled on site and included above)	3 000	8 500	20 000
Supply of *pipework*, fabricated, with valves, and erection with supports, etc. and lagging as necessary	17 000	60 000	180 000
Instruments installed	6 000	20 000	60 000
Electric power wiring and distribution gear	2 500	6 000	15 000
Thermal insulation of plant	1 500	5 250	17 500
Plant installed	78 235	269 540	780 805
Foundations, steel supporting structure, lighting, control room, etc.	16 000	50 000	120 000
Total "within battery limits"	94 235	319 540	900 805

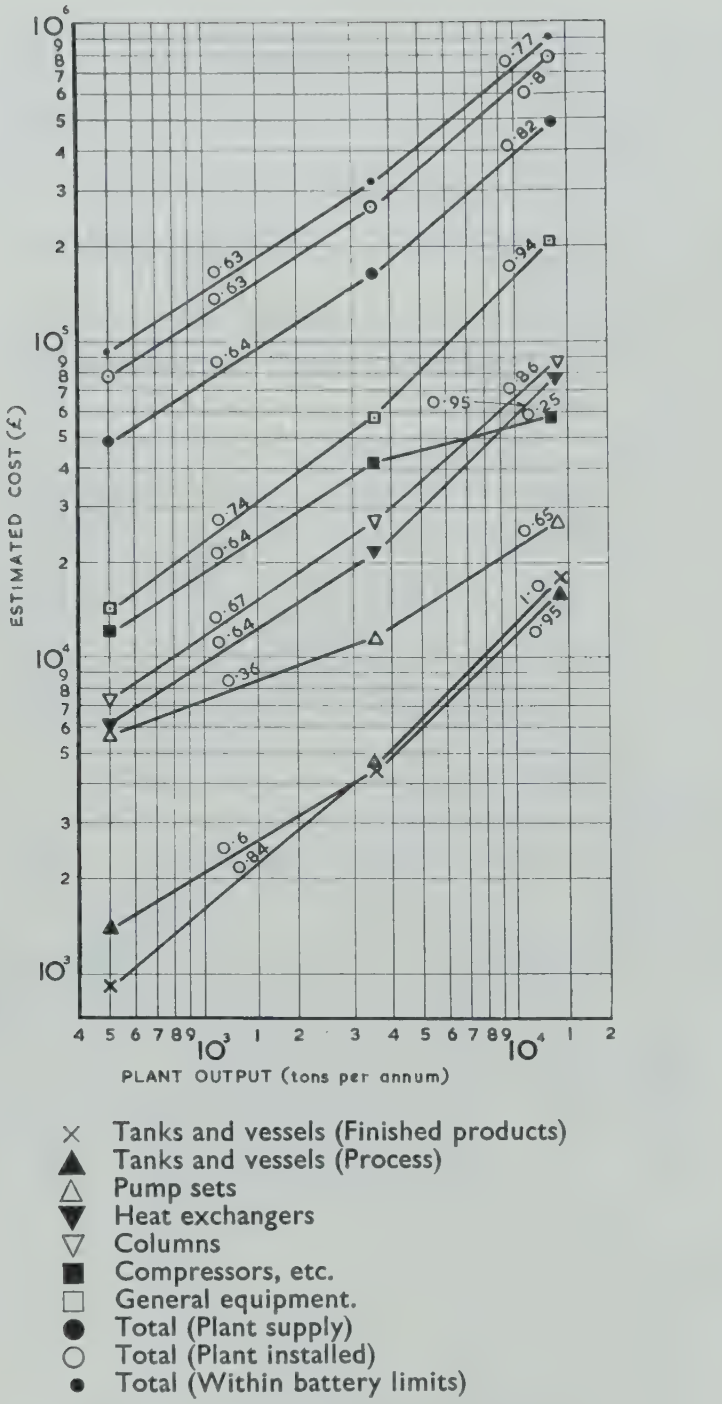


Fig. 9.—*Catalytic vapour phase plant. Construction cost estimate*

operated using a reactor with tubes of diameter and length identical to those proposed for a large-scale plant, together with a recovery system to match, and most of the problems had been solved. However, because further problems were anticipated during scaling-up from the pilot plant, and also to provide an opportunity to test the market for the product, it was thought prudent to construct as the next stage of development a plant with a capacity of about 500 tons per annum although it was not expected to be an economic proposition.

Market assessments had in fact, shown a potential sale for the product of about 3500 tons per annum with a possible expansion to 15 000 tons per annum later. In order to present as complete a picture as possible, of the economics of the proposals, preliminary estimates of construction and production cost were prepared for plants of 500, 3500, and 14 000 tons per annum capacity which will be referred to as plants A, B, and C respectively.

Capital costs

Using the comparison formula previously given, it will be seen that the relationship between plant B which is seven times as large as plant A, is indicated by the exponent of 0·63, and between C and B (4 times larger) by the exponent 0·77, or an overall exponent of 0·7.

To enable an appreciation of the reasons for these results to be formed, some comments on the design considerations of the individual components are given below, following the order given in Table I.

PUMPS WITH MOTORS AND STARTERS

In nearly all cases there are two sets for all applications in plants A and B, and three sets for plant C. This gives greater flexibility for the largest plant although it would have been possible to have only two sets of still larger size with consequent capital saving.

TANKS AND VESSELS

Although the increase required between plants A and B has been achieved by retaining the same numbers and increasing their dimensions, the size of some of the most costly vessels in plant B approaches the practical limit, and therefore the required total capacity for plant C involves an increased number of units.

In the case of the final product storage, a limitation on size was considered necessary because of the danger of polymerisation in larger units. The step between plants B and C is therefore purely an increase in the numbers of units of the same size tanks.

COLUMNS

The increase in capacity from plant A to plant B has been effected by increased dimensions of the same numbers of columns, but in the step from plant B to plant C a limit is reached in the liquid loading on these columns which makes it increasingly difficult, as the diameter is increased, to obtain sufficient length of overflow weir and to achieve a satisfactorily low hydraulic gradient on the bubble cap plates. It was therefore necessary to duplicate some of the columns rather than increase their dimensions.

COMPRESSORS

It was found possible to contemplate using a very small, reasonably-priced, high-speed, turbine-driven, centrifugal compressor for item No. 1 in plant A, and in view of the key nature of this unit a standby set is also included. For plants B and C the range of the type of machine selected for plant A did not extend far enough to meet requirements without multiplication of units. There was, therefore, a change in the design of these units which accounts for a rather larger increase than one would normally expect in the capital cost between plants A and B. For plants B and C, there are two machines in each case, each capable of taking half load.

HEAT EXCHANGERS

For one particular application—the gas cooler following the reactors—a decision was made to install one cooler per reactor, and as will be explained below, the increased capacity of reactors was obtained by multiplication of units. In another case the limit in size of a particular proprietary type of heat exchanger was reached with plant B, and therefore, in total, in the step between plants B and C, the cost was largely affected by multiplication of units.

GENERAL EQUIPMENT

The reactors were nearly at what was considered to be the limit of unit size in plant A. Therefore plant B involved an increase both in numbers and in size of units. The step to plant C was by multiplication of units only.

Capacity of other items such as the vent adsorbers was increased simply by increase of number of units from plant B to plant C. Other less expensive items were increased in unit size and partly numerically.

INSTRUMENTS

The cost of instrumentation obviously depends mainly upon the number of items of equipment rather than the size of the units. There is however, a tendency towards more elaborate instrumentation and more automatic control as the size of a plant increases.

STRUCTURES AND BUILDINGS

As the size of units is increased by scaling-up, so the area and volume occupied in structures or buildings decrease per unit of output. This has been well illustrated by the reported findings of the Central Electricity Authority in post-war construction of power stations.

Production cost

Table II shows estimates of unit production costs for the three scales of operation. These are generalised and are not intended to be a model for absolute values, but all the main variables are taken into account with the intention of comparing relative costs of production at the three different scales as faithfully as possible.

Decisions subsequent to this stage, especially the choice of a specific site for the plant, would mean the revision of all the details using actual factory rates for supply of utilities, and depreciation, overheads, etc. would be determined according to the circumstances ruling and company policy. The additional investment in working capital and the costs of designing and supervising the construction of the plants would also be taken into account.

The total unit production cost in bulk, ex works, shows a considerable reduction as scale of production increases and it is proposed to comment on the contributory factors under the headings already used when stating the principle of " Increased Dimensions ".

CAPITAL

The depreciation, *i.e.* the charges allocated against production costs each year for the recovery of the initial capital outlay, shows an appreciable reduction as the scale of production increases. It is, as shown, proportional to the original investment (ignoring any considerations due to changing replacement values). In some assessments interest on capital employed, or alternatively " opportunity cost ", would be added here. In this case, such an item is not added specifically but would be part of the anticipated gross return on the investment ; it would however be expected to be in proportion to the initial outlay.

EFFICIENCY

Some reduction in consumption of utilities due to the higher efficiency of motors, turbines, pumps, and greater heat conservation of some items although significant is not very great in this particular example. In cases where the proportion of dynamic machinery is higher, the improved efficiency of large units in larger-scale plants would be more important.

The estimates show no saving in raw materials

TABLE II.—*Estimate of Production Cost*

	Scale, tons/annum								
	Plant A 500			Plant B 3500			Plant C 14 000		
	Fixed capital cost								
	£94 235			£319 540			£900 805		
	Per ton	£/annum	£/ton	Per ton	£/annum	£/ton	Per ton	£/annum	£/ton
Utilities									
Steam at £0·8/ton	12·2 tons	—	9·76	11·2 tons	—	8·96	10·7 tons	—	8·56
Cooling water at 3d./1000 gal.	$2\cdot50\times10^{5}$ gal.	—	3·13	$2\cdot16\times10^{5}$ gal.	—	2·70	$2\cdot05\times10^{5}$ gal.	—	2·56
Process water at 1/6d./1000 gal.	6600 gal.	—	0·50	6600 gal.	—	0·50	6600 gal.	—	0·50
Electricity at 1d./kwh.	1530 kwh.	—	6·38	1390 kwh.	—	5·79	1310 kwh.	—	5·46
Fuel oil at £8/ton	0·45 ton	—	3·60	0·41 ton	—	3·28	0·39 ton	—	3·12
			23·37			21·23			20·20
Labour and supervision									
Process operators at £600/annum	—	7200 (12 men)	14·40	—	9 600 (16 men)	2·74	—	14 400 (24 men)	1·03
Supervision	—	2400	4·80	—	3 200	0·91	—	4 800	0·34
Maintenance	—	2400	4·80	—	8 000	2·29	—	22 500	1·61
Depreciation 10%/annum on capital cost	—	9420	18·84	—	31 950	9·13	—	90 080	6·43
Overheads	—	—	5·00	—	—	5·00	—	—	5·00
OPERATING COST	—	—	71·21	—	—	41·30	—	—	34·61
Material A at £30 per ton	0·95 ton	—	28·50	0·95 ton	—	28·50	0·95 ton	—	28·50
Catalyst and miscellaneous chemicals	—	5500	11·00	—	38 500	11·00	—	154 000	11·00
Total Production Cost In Bulk, Naked, Ex Works			110·71			80·80			74·11

usage as the scale is increased, whereas in practice owing to a probable reduction in the proportion of mechanical losses on a larger plant one might expect some slight increase in yield. At this stage of development such possibilities are very uncertain and the more conservative course has been taken of using the actual pilot-plant yields as a basis for all scales of production.

LABOUR

The effect of this is most marked. With the 500 tons per annum plant it was considered that the minimum number of men necessary for controlling its operation was three per shift, but with a modern well-instrumented plant this would rise to four for the 3500 tons per annum plant and six for the 14 000 tons per annum plant.

Overheads including factory management might logically be expected to be reduced per unit of output in the case of larger-scale plants although this does not always prove to be so in practice, and accordingly at this stage overheads are assumed to be a flat rate of £5 per ton for all scales of production.

OVERALL RETURN ON INVESTMENT

The ultimate test in normal circumstances as to whether a plant shall be built, is that of profitability. All other facts and considerations only contribute towards making this final assessment possible. So far, this example has been concerned with the various factors that affect the cost of producing and supplying the product, but now there has to be introduced the income from the sale of the product. In this paper, income and how it is forecast will not be discussed in any detail, but to enable a comparison to be made to indicate the relative profitability of plants at three different scales, a simplified case is assumed where there is a "going" market price equivalent to £100 per ton exworks. It has previously been mentioned also that working capital which is not included here would have to be taken into account.

Plant A (500 tons per annum) would operate at a loss; it might possibly be justified to gain experience and explore the market for the product, but even this is unlikely.

Plant B (3500 tons per annum) would show a gross return of 21% per annum (before taxation) and as such is certainly likely to be of interest.

Plant C (14 000 tons per annum). The gross return would be 40·2% per annum (before taxation) and this plant would probably still show a satisfactory net profit in circumstances less favourable than those anticipated in the assessment.

As stated, the comparisons given above are based upon an assumption that full output will be sold at a "going" market price which is unaltered at the three different rates of production: that is to say demand is infinitely elastic over the range of output considered. This may be so in some circumstances, but it is quite probable that if the product were a novelty, prices would be fixed to suit an estimated demand curve which would be downward sloping; 500 tons per annum could be sold at a higher price, and 14 000 tons per annum would only be sold at a lower price than that used for comparison purposes. In such a case it is still most probable that the rate of return would be higher as the size of plant increases, and that profits would be maximised with the greater output.

We must therefore conclude that given a sufficient demand for the output, the plant on the largest possible scale with its inherently lower unit costs of production is by far the most attractive proposition economically. At the other end of the scale a small plant could be completely uneconomic.

Scaling-Up Methods for Estimating Construction Costs

Engineers concerned with development or construction of chemical plants may often need or be asked to give an " order of cost " estimate of constructing a plant similar to, but of a different output from another which may be in the development stage or already built. Such figures may be required to direct research and development efforts along the most promising channels and towards the most economic investments.

The question always asked on such occasions is, " Is there a short-cut method which will give a reliable answer without wading through a lot of detail ?".

So far we have reviewed some examples of how costs of various types of individual units vary with capacity or output and with the aid of curves have analysed the exponents applicable to a simple comparison formula, over limited ranges of size. Some understanding of the reasons for the relationships which do exist have probably helped in following the example which has been given for three different sizes of a complete plant. It will have been observed that many detailed decisions normally have to be made such as whether units can be increased in output by increasing their dimensions—which is desirable from an economic point of view—or whether the increased output will have to be obtained by multiplication of units which may be necessary because of practical limitations in size, or to obtain flexibility and maximum availability in operation. Possibly too, some combination of increased dimensions with less than proportional increase in numbers of units may be the best solution. Can any conclusion of an empirical nature be formed which will enable this detail to be bypassed ?

Chilton[4] believes that it can, and analyses data published in the U.S.A. to conclude that the " Six-Tenths Rule " does apply to complete plants. This means an exponent of 0·6 in the comparison formula which has already been discussed. A careful scutiny of the data to which he refers and the method employed in correlating the data which is predominantly for petroleum refining plants, indicates that although there must be some error it is probably reasonably small, but the conclusion reached does not appear to be entirely in accordance with the evidence. It is stated by Chilton that the average slope of the curves of all the plants considered including some where increase in output is obviously predominantly by multiple units, is 0·68 and the median is 0·66. He then goes on to say " If we . . . eliminate these higher slopes from our calculation both the average and median values figure out to be 0·62". He appears to be wishing to prove the " Six-Tenths Rule ".

For the average chemical plant of small-to-medium size, the conclusion reached by Chilton is in accord with the author's own experience. As can be seen from the example given in this paper, if increase in output is by " increased dimensions " then it is completely logical to accept and use an exponent of 0·6 when estimating another size of complete plant which is within this range

However, in most chemical plants when scaling up from a small or medium-size plant to a large plant, the effect of multiplication of units is often quite considerable. Assuming there is some split-up available of the actual or estimated cost of the plant which is to be used as the basis for the scaling-up (or down) estimate, then it is well worth the time taken to go over the process comparing technical data with costs for individual groups of items and forming a quick assessment whether each group can be increased in output by increased dimensions or by multiplication of units. The probable cost of each group can then be assessed by using a suitable exponent in the formula (or an average of 0·6), or by simple multiplication, whichever is applicable. The total arrived at in this way is probably the best short-cut method that can be applied in a short time.

Unfortunately, published information on this subject is extremely rare in this country and for that reason an exercise similar to that carried out by Chilton cannot be made. However, from eperience it can be recommended that if time and basic information is not available when scaling-up the cost of a small commercial plant to one quite considerably larger, an exponent of about 0·72 can be used with confidence that it will provide relative costs with a fair degree of accuracy.

Acknowledgment

The author wishes to record his thanks to The Distillers Company Limited for permission to publish this paper.

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Appendix I

Vapour Phase List

Key to Fig. 8.

Item No.	Item
1.	Gas compressor.
2.	Gas preheating furnace.
3.	Fuel oil pump.
4.	Fuel oil heater.
5.	Combustion air fan.
6.	Fuel oil storage.
7.	Reactant compressor.
8.	Reactor.
9.	Heat transfer oil circulation pump 1.
10.	Heat transfer oil circulation pump 2.
11.	Waste heat boiler.
12.	Heat transfer oil expansion tank.
13.	Feed water pump.
14.	Heat transfer oil start-up pump.
15.	Heat transfer oil storage tank.
16.	Gas cooler.
17.	Water storage tank.
18.	Water circulation pump.
19.	By-product scrubber.
20.	By-product circulation pump.
21.	By-product liquid cooler.
22.	By-product cyclone.
23.	Absorber.
24.	De-aerator feed pump.
25.	Absorber feed cooler.
26.	Absorber feed pump.
27.	Balance tank.
28.	De-aerator feed exchanger.
29.	De-aerator.
30.	Vacuum scrubber.
31.	Vacuum pump.
32.	Still feed tank.
33.	Still feed pump.
34.	Still feed exchanger.
35.	Still.
36.	Still reboiler.
37.	Still base pump.
38.	Condenser.
39.	Reflux tank.
40.	Reflux pump.
41.	Product cooler.
42.	Product storage.
43.	Inhibitor storage.
44.	Inhibitor pump.
45.	Vent gas heater.
46.	Vent adsorber.
47.	Chimney.
48.	Nitrogen scrubbe
49.	Nitrogen gas-holder.

DISCUSSION OF PAPERS PRESENTED AT THE FIRST SESSION

Dr. F. SJENITZER suggested a classification of the various methods of scaling ; methods which were fundamentally different but each of which might rightly be called scaling in the sense of increasing, or decreasing, the capacity or size of an operation. He thought that four methods of scaling should be distinguished.

Scaling-up by increasing the number of identical elements working in parallel, as for example in a heat-exchanger containing parallel tubes. Each element received the same material and made the same product. This was often called " multiplication of units ", and was so described by Mr. Dickerson. The characteristic problem of this method of scaling was the correct splitting of feed streams for the elements, which sometimes presented difficulty.

Scaling-up by increasing the size of one piece of equipment while keeping unchanged its design and its function in the flow scheme. This was what was usually meant by "scaling-up", and most of the papers in the symposium were concerned with it.

Scaling-up by choosing another type of equipment, which served the same purpose in the flow scheme but was better adapted for processing larger quantities of material, as when a leaf filter was used for small quantities but a rotary filter for larger-capacity operation. Mr. Dickerson touched on this. It was clear that the scale of operation might have a very pronounced influence on the type of equipment chosen. It was unfortunate that this aspect was not more often discussed in the literature.

Scaling-up by making alterations in the flow scheme. It might sometimes be necessary, usually for economic reasons, to chosen a totally different flow scheme, including other physical or chemical operations or both, as being more suitable for larger-scale operation. This fourth method also deserved to be described more fully in the literature.

Prof. M. W. Thring discussed flames, which were a link between chemical engineering and fuel technology. The flame in a gas-turbine combustion chamber was a good illustration of the point made by Prof. Denbigh about the difference between stirred reactors and plug flow. In particular, the maximum combustion intensity in a gas-turbine combustion chamber was obtained by a combination of a stirred reactor followed by plug flow. The peak of Prof. Denbigh's curve was reached in the first process and the combustion finished with continuous plug flow; to carry out the whole reaction in the stirred condition would be extremely difficult. The only problem in scaling was the big wall-effect, and unless it could be eliminated the right result would not be obtained on a small scale.

An example also relevant to Prof. Denbigh's paper was that of pulverised coal firing in a combustion chamber. He had raised the question of whether there was plug flow or a distribution of residence times, and this was extremely interesting in regard to pulverised fuel combustion. Unless care was exercised the gases would be pushed down the axis of the combustion chamber with a high velocity and the bulk of them would leave the chamber much too soon. There would not be anything like the plug flow which was wanted to give all the particles maximum burning time. At Sheffield University some work was being done on this problem, and it provided a good example of the use of models. In this work they were taking the problem apart in order to use models and studied only the flow distribution, deliberately eliminating combustion and using a cold model with the best similarity conditions obtainable in order to get the distribution of residence times with the kind of combustion chamber which had been developed empirically and which worked quite well.

Fig. 1.—*Flames on three scales*

The third example was provided by luminous radiation. Fig. 1A represents a full-size open-hearth furnace or a full-size boiler and Fig. 1B International Flame Radiation furnace, of perhaps about half the linear dimensions; and Fig. 1C represents the laboratory furnace of about one-fifth the scale of the preceding one. The interesting question was, why build the comparatively big furnace (B), which burned a great deal of fuel, in order to study (A), when it ought to be possible to get the results which were wanted with (C).

The answer was that it was not possible to get the results required with (C). The work done on (B) showed that the relation between soot and luminous emissivity depended on the length of the flame. If a curve were plotted for soot against distance along the flame, the curve came down as the length of the flame was shortened. Thus one might derive a formula

$$\epsilon = 1 - e^{kls}\,(1 - \epsilon_N)$$

where ϵ was the total emissivity, l the flame thickness, s the soot concentration and ϵ_N the non-luminous emissivity. It might be said that the soot concentration in this flame was a function of the kind of fuel, the distance of the point concerned expressed as a portion of the length of the flame, and the length of the flame as the final parameter,

$$s = f_1\left(\frac{C}{H}\right) \cdot f_2\left(\frac{l}{l_f}\right) \cdot f_3\,(l_f).$$

All the work which had been done on (B) gave a formula of that sort. If the fuel input rate was doubled with the same size of nozzle, there would be the same length of flame, and there would be the same soot curve and the same luminous emissivity. That was with furnace (B), where there was a length effect, but with (C) one did not obtain that at all. It might be expected from extrapolation that there would be almost no soot from the small flames, but that was not so; there was about the same soot concentration from the very small flames as from the big ones, so that something had gone wrong. They did not know why, but it looked as though in scaling from (C) to (B) it was time which mattered and not length. Probably that was because of the processes which went on in the early part of the flame, when the soot was made. In this case not enough was known about scaling-up to predict from (C) the results with (B), and therefore with (A). It illustrated, in other words, the complexity of the scaling problem.

Mr. R. P. AYERST said that Prof. Kramers and others[1] had studied the rate of mixing in stirred tanks and had tentatively concluded that the rate of mixing would be proportional to the linear dimension with the same agitator tip speed. This led to the conclusion that with a large tank it would be necessary to have either a very long time of mixing or a very high power consumption. Could Prof. Denbigh say what ratio of blending time to retention time was reasonable to allow the subject to be treated in the way suggested in his paper?

Mr. F. W. EDWARDS said that in Table II of Mr. Dickerson's paper the figure for maintenance was 5% of the machinery cost given in Table I for each of the scales of operation. It would be of interest if the author would indicate how much this percentage figure varied for different types of chemical plant. Did he regard 5% as the best figure to take in the absence of any other information?

Referring to the "six-tenths rule" or, the more accurate and convenient "two-thirds rule" for scaling-up the cost of a complete chemical plant, Mr. Edwards thought that a word of warning was desirable, because, while chemical engineers frequently needed a short-cut method for scaling-up chemical plant costs, they should be aware that there could be a variation from 0·6 to 0·7 with certain types of plant, sometimes higher with a multiplicity of units but also sometimes very much lower. Reference to this has been made by Edgeworth Johnstone.[2] He concluded however that the two-thirds rule could be accepted in the majority of cases. For a complete phosphoric acid plant, ranging from 10 to 100 tons of acid a day, the rule appeared to hold extremely well. It would be interesting if Mr. Dickerson would indicate how well the rule held for plants which might be described as predominantly solid-handling and incorporating such items as conveyors, screens, and grinders.

Dr. F. V. A. ENGEL said that Mr. Edgeworth Johnstone and Prof. Thring presented some of the major problems regarding dynamic similarity in a thought-provoking way. Writing some twenty years ago Edgeworth Johnstone had remarked that a new era in chemical engineering design might have been started by G. Damkoehler. A glance at the references and at the work done by various authorities might show that this prediction had not yet been realised. The symposium corroborated this view, since most of the papers were suggestive in character and did not completely solve the major problems.

In their present paper Johnstone and Thring presented some major problems in a very apt way, giving emphasis, for example, to the investigation of model elements. Some of the statements made, however, should be qualified. Dr. Engel proposed to concentrate on one major issue, which might explain why progress had been comparatively slow. This was related to the concept and interpretation of the meaning of the non-dimensional groups of dynamic similarity.[3]

His thesis[4] was that a number of the ordinary operations of algebra became meaningless when applied to the true non-dimensional groups. An example was afforded by the interpretation of the Navier-Stokes equations, which rightly included a term related to unsteady flow. The authors argued that dimensionally the velocity was equal to length divided by time, and said that a crucial term for unsteady conditions should be omitted from further consideration. Following a similar argument, it would be justifiable to omit the Reynolds number as meaningless, as it was composed of two terms which cancelled dimensionally. Similar procedures had been adopted by other authorities, some of them even stating that the Reynolds number was merely another version of the Froude number. This went against an important axiomatic rule, that the non-dimensional groups formed entities in which many of the usual mathematical operations were meaningless. The Strouhal number, which was an important flow criterion cancelled by the authors might have in some cases the same importance as the Reynolds number.

Power function relationships also had to be considered carefully. Products of powers of non-dimensional groups were valid only over restricted ranges. Applying such terms to extrapolations beyond the limits obtained by the pilot-plant investigations and making use of those results in the full-scale plant might result in a serious misinterpretation of the process itself, leading to wrong dimensions for plant design. He suggested therefore, that a very critical view should be taken regarding the application of the true non-dimensional groups of dynamic similarity to the scaling-up of chemical plants.

The authors rightly drew attention to the so-called mixed régime in which several groups were significant, which necessitated scaling-up according to different laws, the Reynolds law and the Froude law. In model research in civil engineering everyone was familiar with the so-called scale effects, but the situation was not so simple as the authors thought. One could not consider, say, the Froude group as the dominating factor and the Reynolds number as merely resulting in a correction factor. The Telfer diagrams referring to ship resistance problems, and some of Dr. Engel's own investigations on Venturi weirs showed considerable discrepancies if only one of the laws, either the Reynolds or the Froude law, was considered to be predominant. Model investigations on a single pilot plant, due to this so-called scale effect, might not produce satisfactory results. This depended, of course, on the accuracy limits which were expected from such investigations. A family of models should be analysed to predict with sufficient reliability the full-scale plant in its dimensions and performance.

He therefore recommended a critical review of the true non-dimensional groups of dynamic similarity and the related admissible mathematical manipulations. A further step referred to a thorough study of the problems related to scale effect, which meant a disentanglement of various influencing factors over considerable ranges of non-dimensional groups.

Mr. J. P. Asquith, commenting on the reference in the second paragraph of the paper by Mr. Edgeworth Johnstone and Prof. Thring to the fact that when the electrolytic aluminium process was scaled-up the problems associated with the small-scale experiments "disappeared as if by magic" on the large plant, said that this was not unique. He believed that it was virtually impossible to make pigment zinc oxide in the laboratory, and that was equally true of the lead oxide which was manufactured in the first stage of making red lead. In desilvering lead by the zinc process much higher enrichments were obtained very much more easily in a 300 ton pot than in a 10 ton pot.

It was too easy to say "This having happened in these cases, it will happen in all", because then it would be possible to scale-up a great many processes without any worry. The authors had tantalised their readers by mentioning this point and then ignoring it.

Referring to Mr. Dickerson's paper, Mr. Asquith proposed to confine himself to the problem of the short-cut method for getting out the scaled-up price as well as the scaled-up design. One of the facile methods was to do this in relation to the annual sales value. This was based on the theory that annual sales value was related in a simple arithmetical way to the capital cost of the plant. Mr. Dickerson seemed to have disposed of that. He had given not the sales value of his product but a works equivalent price of £100 per ton at which sales should be profitable. If his outputs were multiplied by that, the figures obtained were: for Plant A, £50 000; for Plant B, £350 000, for Plant C, £1 400 000. Those compared with his pre-design estimates of £94 000, £319 000, and £900 000 respectively. It was probably fortuitous that the middle one was in the right field.

It was Mr. Asquith's experience that one of the most realistic aproaches to the quick price scale-up was by the use of the percentage breakdown method, which was only a variation of, though a refinement of, Edgeworth Johnstone's bulk factors.[2] If Mr. Dickerson's Table I were broken down by expressing the value of each item as a percentage of the total, some interesting features emerged. The most interesting one was to take the percentage breakdown of Plant A and then take the intermediate sub-totals of Plants B and C, £169 790 and £488 305. The percentage for Plant A of the sub-total to the final total was 51·19% If the figure of £169 790 was increased on the basis of 51%, which was the breakdown of Plant A, and not on its own breakdown figure, which was 53%, it would give an estimated total for the whole of Plant B of £332 000, which was within 4% of the author's figure. If the same were done for Plant C it came within 6% of the author's figure.

The author deserved their thanks for giving them one more set of figures with which to carry out this exercise. To break it down on the basis of the individual items brought out the point made by Mr. Edwards about the need for care, because if one took a summary calculation based on the first half-dozen headings and used the percentage of the total make-up from Plant A and the £ values for individual items from Plant B one would get some very erratic figures. Taking it on the pumps would give a total figure for Plant B of about £200 000, while taking it on the process tanks would give a figure of about £300 000, and on the finished products tanks a figure of £450 000, compared with the figure given in Table I of about £319 000.

Mr. **Th. W. Willemse** referred to a statement in the paper by Mr. Edgeworth Johnstone and Prof. Thring which seemed to him to be not quite correct. In the left-hand column on p. S5, in the derivation of the "generalised dimensionless equation" for the pressure loss in fluid flow, it was stated that "Dimensionally $v = L/t$, hence $L/vt = 1$".* Obviously this applied to any dimensional formula appearing in this and similar equations. It was the very feasibility of writing physical equations in terms of variables the dimensional formula of which was unity which expressed the basic fact that the description of the laws of nature was independent of the choice of the size of the units.

What seemed to be meant in omitting L/vt from the equation referred to was the expression of the fact that strict kinematic similarity was not required in scaling up; *i.e.* a change of scale did not require a proportional change in velocity. In other words, the way in which the phenomenon in question was enacted was independent of the velocity, as long as no account was taken of dynamic effects, *i.e.* as long as one did not bother about Reynolds or Froude dimensionless numbers for instance. That meant that one was free in the choice of the time-scale, L being fixed beforehand, v was not yet fixed. There was freedom in the choice of the time-scale, since no direct connection existed between time and the spatial dimensions. The reverse might be true if relativistic velocities occurred, as might be the case with high-voltage particle accelerators.

Mr. **A. L. de Gelder** said that he had not found anything in the paper by Mr. Edgeworth Johnstone and Prof. Thring about boundary conditions. The authors drew attention to the fundamental differential equation which governed the field of flow of a certain apparatus. Differential equations, however, inevitably demanded boundary conditions, the latter being the expression for the problem in hand, while the differential equations were not restricted to a special apparatus. An apparatus was in the first place defined by its boundaries, the field being of importance for what it was desired to do with the apparatus.

Mr. de Gelder wished to show the importance of the boundary conditions. He took as an illustration an apparatus with a curved deep channel and assumed that it was desired to examine properties of the flow in the channel in connection with, for example, heat transmission to the outer wall. It was tempting, as indicated by point 2 of the authors' principles, to take a full-size replica of a section of the channel. There would be a curved pipe with a quadratic section, and the velocity distribution, uniform at the entrance, would be distorted after passing the curve in such a way that the largest velocity would be at the outside and a retarded layer at the inside. The eventual conclusion drawn from this experiment would, however, be quite disastrously wrong if applied to the original. In a deep curved channel the distribution of the velocity, and therefore the retarded layer, would, owing to secondary flows, be just the inverse; the retarded layer would be at the outside and the highest velocity at the inside.[5]

Another example illustrating the importance of the boundary conditions was the roughness of the wall. At a certain value of Reynolds number it was well known that the drag coefficient of a sphere in a uniform stream had a sudden fall to a lower value, when the boundary layer was completely detached at the rear side. By fixing a thread over the surface of the sphere, it was possible to influence considerably the value of the Reynolds number for the detachment; *i.e.* without changing the fundamental equations the problem was strongly influenced by changing the conditions at the boundary.

Mr. de Gelder pointed out that the Navier-Stokes equation applied to a fluid of changing density as in non-isothermal flow. In this case an additional equation was required to deal with the heat liberation and heat transfer. The Navier-Stokes was essentially the equation for compressible flow.[6]

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* This refers to the preprint. The authors have taken the remarks of Mr. Willemse into consideration in the published version.—*Hon. Ed.*

Mr. P. J. HOFTYZER remarked that from Prof. Denbigh's paper he had learned that the best reactor for a given process could be evaluated from a knowledge of kinetics, so that it was not found by gradually enlarging the size of the laboratory apparatus. It was remarkable that a paper on the subject of scaling-up did not contain any linear dimensions. The conclusion could also be drawn that the reactor which was best suited to the process could not be chosen if the kinetics of the reaction were not sufficiently understood. It should also be stated that a model of the reactor might still be useful for the study of technological problems, such as, for example, the prevention of foaming, and bubble formation on the coming into contact of two fluids.

Those principles might be applied to a reaction mentioned by Prof. Denbigh, the catalytic oxidation of ammonia. In some laboratory experiments, an optimum contact time had been found. This might be due to a degradation reaction, namely the decomposition of nitric oxide, as suggested by Prof. Denbigh, but it might also be caused by the thermal recomposition of ammonia at the walls of the laboratory apparatus. In the latter case the optimum contact time as found in the laboratory experiments would not apply for the full-scale apparatus. Here also a knowledge of the kinetics would be necessary for the design of the technical reactor.

Dr. A. KLINKENBERG said that in the summary of the paper by Mr. Edgeworth Johnstone and Prof. Thring it was stated that there were two methods of deriving similarity critieria, by dimensional analysis and from differential equations. Those two methods were of wide applicability and somewhat difficult. There was, however, a much simpler method, though not of such wide applicability. There were cases where the entire process might be described by ordinary equations, not differential equations, and it was a matter of simple algebra to transform them to the dimensionless form. That automatically gave the similarity criteria.

On p. S4 the principle of similarity was discussed in relation to scaling-up. It seemed to him that the principle of similarity, though very closely related to the problem of scaling-up, covered slightly different ground and that the fields of the two did not entirely overlap. It could be said that two processes were similar if they were governed by the same equations and the same type of boundary conditions and if the variables occurring in those equations had the same numerical values. With that general definition he had reduced the process to a mathematical equation, and this meant that he would also call two processes from different fields of physics similar or, it might be, two chemical engineering processes such as heat transfer and mass transfer, if they correspond to the same equations and if all the variables were the same. Indeed—although this was not scaling-up, but the science of models—there were many cases where models had been made in fields other than that in which one was primarily interested. Such a comparison between a heat-transfer model and a mass-transfer technical plant was not scaling-up in the sense of the Symposium but would be similarity. He would like to know whether or not Prof. Thring agreed with him that the principle of similarity should be extended to cover these cases.

When devoting attention in this way to the underlying mathematics, it became simpler to understand what was meant by distorted models. It was stated on p. S4 of the paper that " Models and model elements may be either regular or distorted. A distorted model has different scale ratios along different co-ordinates. Thus there is a considerable latitude in the shape of small-scale unit that may represent a given prototype while conforming to the requirement of geometrical similarity ". It seemed to him that readers might have difficulty in interpreting this paragraph, because no guidance at all was given as to when distorted models could be used. It had been said that similarity had something to do with, and included, geometrical similarity, but now an exception was being made for cases where one might drop one of the arguments and say that, after all, geometrical similarity was not necessary and a distorted model could be used.

If a return was made to the fundamental mathematics, and if it was said that systems were similar if the underlying mathemetics were the same, in the way he had explained earlier, it became much easier to understand. It might be that in the underlying mathematics the full geometrical similarity was not required. A case in point was the equation for pressure drop in a flow in a straight circular pipe :

$$\frac{\Delta p}{\frac{1}{2}\rho v^2} = f\left(\frac{l}{d} . Re\right)$$

There were three determining dimensionless groups: pressure drop/kinetic energy, length/diameter, and the Reynolds number. If the pipe was very long, so that entrance effects could be neglected, the pressure drop would have to be proportional to the length, so that for long pipes this equation degenerated to a simple form:

$$\frac{\Delta p}{\frac{1}{2}\rho v^2} \cdot \frac{d}{l} = f(Re)$$

It was known from physical considerations that pressure drop must be proportional to length, so that two of the groups could occur only in product form. For flow in long pipes it could be said that, as long as l was very large with regard to d, the geometry of the pipe did not enter any more, so that all flow through long pipes for equal values of Reynolds number was similar, whether the pipes were geometrically similar or not. In this case, therefore, one had a distorted model.

Another instance was the flow through sand beds, for instance the flow in a so-called oil reservoir. Here models could be made where the grain-size of the sand was not reduced proportionately. It was possible to do model experiments on the flow of oil underground by reducing the size of the oilfield to one-thousandth of what it actually was without reducing the sand size by a factor of 1000, which, of course, would be an impossible model.

Authors' Replies

Prof. M. W. Thring, replying on behalf of Mr. Edgeworth Johnstone and himself, said that the first type of similarity about which Mr. Sjenitzer spoke, involving a number of elements, was straightforward, and the only problem, as Mr. Sjenitzer said, was to see that each element received the same treatment. The second type was that which had been discussed that morning, but Prof. Thring did not think that the other two really came within the confines of the Symposium because they were not instances of where a study on the smaller scale gave information about the larger scale. They were really cases where one begged the question and assumed that one already knew the design of the large unit and the small model was not used to find out anything about that part of the system.

Dr. Engel had pointed out that unsteady flow was not considered. Rather, it had been eliminated by the way in which the Navier-Stokes equation had been treated, but it was a very good example of the usefulness of deriving one's dimensionless numbers from that equation. It was only necessary to take the ratio of the first two terms on the left-hand side and the Strouhal number was obtained immediately, except that time was replaced by the reciprocal of the frequency of the oscillations for the unsteady processes which occurred. The Strouhal number was then obtained as the ratio of two velocities. That point came out clearly from the equation; much more clearly than by deriving it from dimensional analysis.

In that connection reference might be made to the remark of Mr. Willemse about the elimination of $v = L/t$. Prof. Thring agreed that the only significance of putting the l in was that if there were two systems, a small one and a big one, there was a length scale between them, and a time scale, and a velocity scale, and it was not possible to choose all three independently. If the length and time scale were chosen, then the velocity scale was fixed. That was all that the authors had meant, and he agreed that as soon as the velocity of light came into it, as in relativity systems, or that of sound, where the Mach number was appreciable, the velocity ratio was tied and the time scale was fixed by that alone; it was no longer possible to make the Reynolds number what one wanted it to be and scaling effects were large and much worse in those cases. Fortunately, it was normally possible to neglect them or to cheat and eliminate the part where the Mach number was high, as was done in burner systems.

Mr. Asquith's remarks were very relevant, but probably a detailed study of the zinc oxide and lead oxide systems to which he referred would indicate the relevant processes, and therefore it would be possible to work out which appropriate scaling effect had been neglected, and then one could again cheat and make the smaller model work by putting in heat at the walls or by making the model different in some other way.

Dr. de Gelder referred to the boundary wall. It was true that it was not mentioned in the paper. Wall roughness and wall effects should be considered, but if one had complete similarity there was no difficulty, because the dynamic similarity allowed for them. It was only if one wanted to cheat, as Dr. de Gelder did in his example of incomplete similarity, that it was necessary to consider the wall effect separately.

He had to accept Dr. Klinkenberg's views, which were very sound. On the point about differential equations against integral equations, Prof. Thring thought that if one went from the differential equation to the integral and based the similarity on that there was no real difference. It was when going from the integral equation to extrapolate the values of the dimensionless parameters that difficulties were encountered. If one used a certain power of the Reynolds number and then a certain value of the Reynolds number in the model, and a 100 times bigger value in the full system, the extrapolation might not hold, so that care must be exercised in going from differential to integral equations.

On the question of similarity versus scaling-up, he agreed with the distinction which had been drawn. He and Mr. Johnstone had used the term " analogue models " for the systems about which Dr. Klinkenberg had spoken, where there were two different physical quantities obeying the same differential equation. Prof. Thring thought that that was the correct use of the term " analogue models ", although sometimes something was described as an analogue model when it was just an electric circuit which was solving a differential equation by approximation and it was not a true analogue at all.

Dr. Klinkenberg's example of the long pipe as a distorted model did not seem to be a very good one, because if the pipe was filled with round balls, when the pipe was distorted and made shorter all the balls would become ellipsoids. In other words, a long pipe was not a distorted model of a short pipe, but merely two short pipes stuck together. This was a question of nomenclature.

The grain size in sands was certainly the type of case where one dimension, that of the sand, could be neglected, and it could be assumed that it governed the system as a whole and was not something which had to be scaled. It was a very good example, because it was a type of similarity which was very frequently used.

Prof. K. G. Denbigh thanked Prof. Thring for several very interesting examples of the distinction between tubular and stirred tank reactors in the industrial field.

Dealing with Mr. Ayerst's reference to the criterion of mixing, he said that in most stirred tank reactors he did not think that there was any great difficulty in getting a fair approximation to perfect mixing, particularly if the viscosity of the liquid phase was not too great. He had studied a continuous stirred tank reactor on an industrial scale. The mean residence time in the first tank had been about 10 min. In a mixing test it was filled with water, a stirrer was started at the normal speed and a little dye was poured in. So far as could be seen visually, that dye had been uniformly distributed throughout the vessel in a period of 8 sec., or someting of the order of 1/80th of the mean residence time. The conditions needed to get such good mixing were, he thought, that the number of revolutions of the stirrer should be large within the mean residence time in the reactor, or in other words there should be many revolutions within the half-life of the chemical reaction. The very fact of using a large vessel meant that the reaction was relatively slow, and therefore it was not difficult to get good mixing within the half-life.

Mr. Hoftyzer had aptly pointed out that no linear dimensions were mentioned in a paper dealing with scaling-up! The paper, of course, dealt with only one of the aspects of the scaling-up problem. Prof. Denbigh had taken as his subject the choice of batch or tubular or stirred tank reactors. Having made that choice, the next step in the scaling-up procedure would be to consider the actual size of the vessel needed for the particular job. This question of calculating the size had been very well covered in the literature. In the tubular reactor the assumption of plug flow was frequently used. It was not a bad approximation with turbulent flow, but it was not so good with laminar flow. Even with laminar flow in a tubular reactor, however, it was possible to calculate that the performance, measured by output, was not more than 20–35% less than it would be if there were perfect plug flow.

Mr. Hoftyzer had correctly pointed out that it was necessary to have a knowledge of the kinetics before it was possible to apply the kind of considerations dealt with in his paper. That was entirely true. In many instances, a short time spent on a laboratory investigation of the kinetics would pay handsomely in the final design of the large-scale plant, and that laboratory study should certainly be done in any case.

Prof. Denbigh commented that in the process of dimensional analysis, the variables were gathered together into dimensionless groups, π_1, π_2, π_3 and so on (*e.g.* the Reynolds number, Nusselt number, etc.). According to the π theorem in dimensional analysis, the relationship between these variables was any function whatever, so that one could write $\pi_1 = f(\pi_2, \pi_3 \ . \ . \ . \ )$ In chemical engineering it was conventional to take this function to be a power function, but there was no *necessity* for this. A typical example was the Dittus-Boelter equation where the Nusselt number is taken to be a power function of the Reynolds and Prandtl numbers. There was no logical necessity for such a functional form. Exponential or logarithmic functions of the Reynolds and Prandtl groups would be just as good in principle and might be more suitable in particular applications. It had to be found by experiment which type of function is the most satisfactory in a given range of the variables. This was not to say that power functions are not suitable; simply that one had the freedom to use other types of function if they seemed preferable.

Mr. R. G. Dickerson said that Mr. Edwards had raised the difficult problem of comparing maintenance costs at different scales of operation, and, having shown that the figure taken in the paper was 5%, asked what was the variation of that figure for different types of plant. Mr. Dickerson could only say that he did not fully know. It could vary according to the attitude of management, company policy, and, of course, it usually increased as an annual percentage over the life of the plant; in the first year or two it might be very little and in the later years it might be quite high. Investigation into historical data with changed cost levels, rarely permitted the isolation of the effect of such variables; it was very difficult to obtain some uniform results from analyses of costs, from which forecasts for specific cases could be made with hope of reasonable accuracy. It should be appreciated that the percentage was an average one for the purpose of comparison of the schemes in question.

It had been interesting to have Mr. Edwards's general confirmation of the use of the "two-thirds rule" in scaling up, and his warning that it could be higher or lower. The separate curves given in Fig. 9 (page S22) for the constituent parts of the complete plant showed that there could be this variation, and, according to the combination of various types of equipment, the overall exponents might be expected to vary. Mr. Dickerson had emphasised that he liked to consider groups of similar equipment, to take the technical information together with the costs on which the forecast or estimate was based and to arrive at sub-totals for those individual groups. It was still only an approximation, but by taking smaller units it would be possible to visualise the position more easily and arrive at a total in which one would have more confidence.

On the question of how a rule of this kind applied to solids handling plant, he stated that he preferred to avoid its use when dealing with equipment of that nature. Taking conveyors or crushers as examples, if one plotted price against capacity (other things being equal), the curve would go up in steps. The main reason for this was that with a conveyor, for example, the driving unit might be the same for all capacities within a certain range, and then there would be a change to the next size. In other words, the maker would employ a restricted range of standards as far as possible. When the information was available, it was desirable to draw such a curve rather than use an exponent. In general the use of an exponent for complete plants would give satisfactory results providing there were a sufficient number of units of different size and type to provide compensation for the factor of stepwise progression in individual items.

Mr. Asquith commented upon certain ratios or relationships provided by the breakdown of the estimated cost of various groups of items or elements of the total cost from Table I (page S21).

In Mr. Dickerson's company, actual costs of plants constructed were normally analysed to provide "factors" for pre-design estimating and no doubt other companies did the same. It was important that a consistent method of costing, analysis, and application of such factors was developed and adhered to. Assuming that information was available to permit reasonably accurate estimating of the cost of plant items, one could then apply the overall bulk factors as derived, to give a fair indication of the probable total cost, assuming that practices in design, purchasing, field methods etc. were similar to those for the projects from which the factors were developed. This method was particularly suitable for giving quick results, and had often been described in the literature on the subject.

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INFLUENCE OF DESIGN VARIABLES ON CAPACITY AND EFFICIENCY OF ROTATING DISC CONTACTORS

By Dr. G. H. REMAN*

SUMMARY

In the design of commercial installations involving the unit operation of extraction, particularly for new applications, the necessary data are generally based upon test results obtained in small laboratory apparatus with the liquid system concerned. For the rotating disc contactor this procedure has always been followed. It is evident that a very sound knowledge is required of the effect of scaling-up upon capacity and efficiency.

It will be shown that, besides the properties of the liquid system, the energy input per unit of contactor volume is the main variable determining the capacity of the contactor. The ratio of the flow rates of the dispersed and continuous phases, which is another factor influencing the capacity to some extent, has to lie in a certain range for stable operation of the apparatus.

From the aspect of efficiency, the energy input per unit of contactor volume is also an important variable. It affects both the mass transfer rate and the mixing of liquid between the compartments. As regards to mixing between the compartments, the flow rates of the phases are also of importance for the efficiency.

Introduction

Correlation formulae for the design of a rotating disc contactor would have to express correctly the influence exerted by structural factors of the contactor and properties of the liquid system on the efficiency and capacity. It is found, however, because of the complexity of many liquid systems, that this ideal is difficult to fulfil. Hence, the aim in all development work on the rotating disc contactor has been to arrive at correlations which describe the influence of the structural factors on capacity and efficiency, but which exclude the properties of the liquid system. Once these correlations have been found, a few experiments in a laboratory contactor with the liquid system in question will provide the data necessary for sizing technical contactors.

This approach to the problem leads to relations between dimensional groups, and it might therefore appear that the relations are only of limited value for scaling-up. However, in developing the correlations and design rules, the combinations of structural variables used were chosen on the basis of the phenomena for which the dimensionless groups are more or less well-known. Since full attention was paid to the effect of scaling on these phenomena, it will be clear that the argument against using dimensional groups is of little practical significance.

In this paper the capacity and efficiency relations are stated and discussed after a brief description of the principle of the rotating disc contactor.

Principle of the Contactor

The contactor (Fig. 1) consists of a number of compartments formed in a vertical cylindrical shell by a series of stator rings, with a rotating disc centered in each compartment and supported by a rotating shaft.

The lighter liquid enters the contactor at the bottom and flows upwards counter-currently to the descending heavier liquid, which enters at the top. One of the liquids is dispersed. The size of the droplets is controlled and maintained in each compartment by employing the appropriate rotor speed. No interstage settling and redispersion is practised. Flat rotor discs and stator rings without any protrusion are used, since they create uniform shearing conditions and assist in obtaining a small spread in the size of the droplets.

The flow pattern of the liquid in the compartments is such that the desired counter-current flow of the dispersed and continuous phases occurs. This flow consists in the first place of a rotation of the whole mass of liquid; super-

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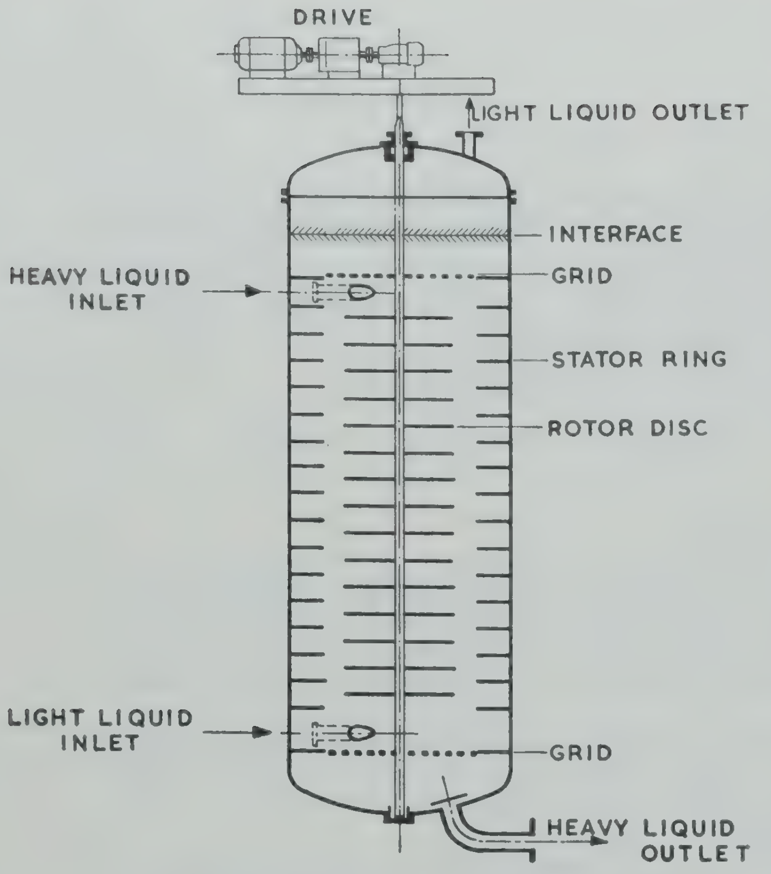


Fig. 1.—*Rotating disc contactor (Interface in top compartment)*

imposed on this rotation there is a slower movement of the liquid from the shaft towards the wall of the contactor in the vicinity of the rotor discs, and from the wall back towards the shaft in the vicinity of the stator rings. The resulting flow of the liquid is thus toroidal. In each compartment two vortices in opposite senses occur ; they are geared together to form a complete vortex.

The energy transferred from the rotor discs to the liquid further creates a fairly uniform turbulence.

Correlation of Capacity Data

Initial data obtained with standard test systems in contactors ranging in size from 2 in. to 8 in. indicated the following facts, some of which are more or less obvious. There is :

(1) a decrease in capacity with increasing rotor speed and increasing diameter of the rotor discs,

(2) an increase in capacity with increasing diameter of the opening in the stator discs,

(3) an increase in capacity with increasing compartment height, and

(4) a decrease in capacity with increasing ratio of the flow rates of the dispersed and continuous phases.

The approach which eventually led to a successful correlation of these data (and others obtained later in contactors of appreciably larger diameter) started from the obvious fact that the drop size of the dispersed phase will play a preponderant role in determining the capacity. A rotating disc contactor in principle deals with an agitated emulsion ; hence it follows that a relationship describing the size of the drops in an agitated emulsion was needed.

Hinze[1] has shown that in a turbulent field of flow the size of the drops in an emulsion is determined by the energy input per unit mass of the emulsion as well as by the physical properties of the liquid system. Since the flow of the liquid in the compartments of the rotating disc contactor may be assumed to be turbulent, application of the above concept appeared obvious. The energy input can readily be expressed in the structural variables. The relation for the energy input per disc in the rotating disc contactor, P, is similar to the well-known formula for other impeller types :

$$P = \text{const.}\ \rho N^3 R^5$$

so that for the energy input per unit mass we obtain

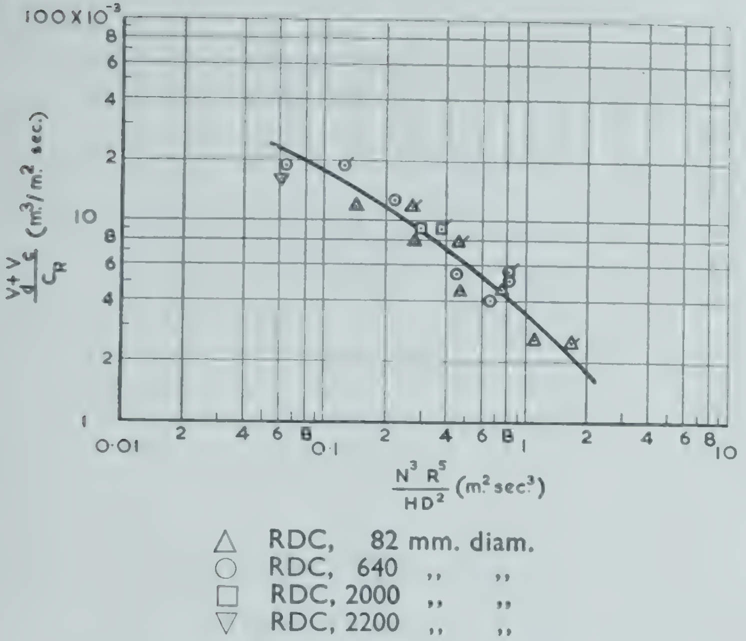
$$\frac{P}{HD^2\rho\ .\ \pi/4} = \text{const.}\ \frac{N^3R^5}{HD^2} \qquad . \quad (1)$$

Thus for a given liquid system we may expect the quantity N^3R^5/HD^2 to be an important variable in determining the drop size of the dispersed phase, and consequently for correlating capacities of rotating disc contactors.

There now remains the problem of finding a suitable expression for the influence of the flow pattern in the compartments and of the restrictions of the contactor shell caused by the stator and rotor discs on the settling of the drops and hence on the capacity of the contactor. The region near the planes of the discs, where settling of the drops occurs in a liquid flowing almost horizontally, is critical for settling. In the regions of the stator disc or the rotor disc, conditions for flooding may occur. For correlation the easiest method to deal with these effects is to correct the observed capacities by dividing them by a factor equal to the ratio of the minimum cross-section and the free cross-section of the contactor shell.

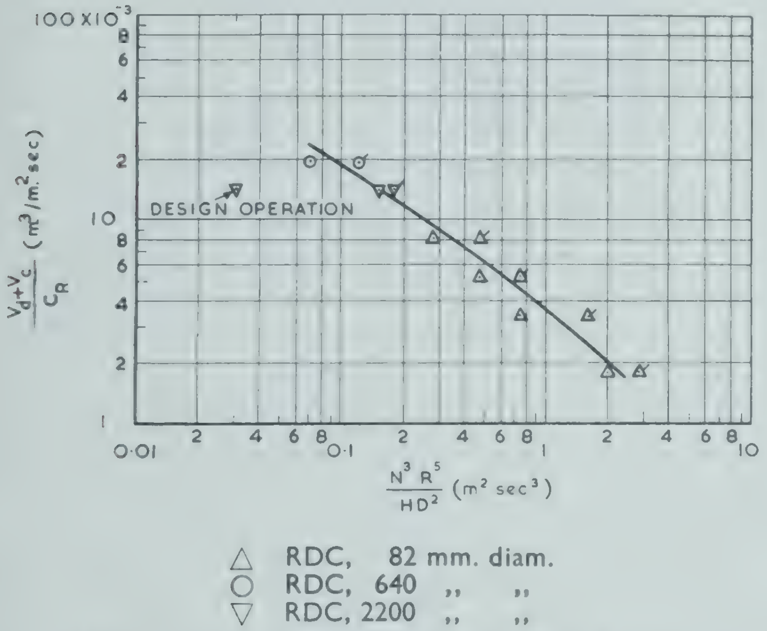
The success of this method of correlation for scaling-up the capacity of the rotating disc contactor can be judged from Figs. 2A and B.

The results given in these figures are valid for a constant ratio of the flow rates of the dispersed and continuous phases. As already mentioned, the capacity decreases with increasing ratio of flow



Open points, no flooding. Flagged points, flooding

Fig. 2A.—*Flooding curve for system Lub-oil distillate (220 seconds Redwood No. 1 at 140° F.)—Furfural; Furfural as continuous phase. Ratio furfural to oil = 2·0*



Open points, no flooding. Flagged points, flooding

Fig. 2B.—*Flooding curve for system de-asphalted residue (750 sec. Redwood No. 1 at 140° F.)—Furfural. Furfural as continuous phase. Ratio furfural to oil = 3·4*

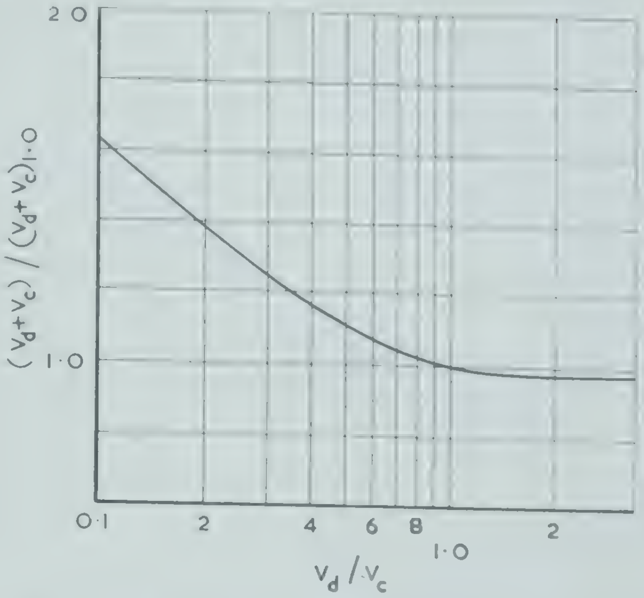


Fig. 3.—*Influence of phase ratio on maximum capacity*

rates. This phenomenon is a general characteristic of the counter-current flow of a dispersed and a continuous phase and can easily be deduced mathematically. For the rotating disc contactor the relationship between capacity and phase ratio is nearly the same as that for spray extraction columns (see Fig. 3, which applies to both types). This relationship only holds if the properties of the liquid system are not altered by the change in phase ratio.

At phase ratios higher than about 3, where hold-up of the dispersed phase at flooding becomes larger than about 50%, the hold-up becomes so high that the concept of the normal hindered settling of the drops breaks down. Coalescence of drops and large-scale irregular flow in, and between, the compartments occur. Irregular flow reduces the efficiency. Operation of the contactor becomes more or less unstable in this region and the scaling-up of the capacity therefore becomes unreliable. For these reasons the design and operation of the contactor should be for ratios of dispersed and continuous phase flow rates of less than 3·0, even though this figure hardly forms a limit to the applicability of the contactor.

Correlation of Efficiency Data

Data obtained with standard test systems,* in contactors ranging in size from 2 in. to 16 in., indicate the following trends for the efficiency, the latter being expressed in terms of stages per metre of contactor height (see Figs. 4, 5, 6, 7, and 8):

(1) In general the efficiency increases with increasing rotor speed. In certain cases, however, the efficiency passes through a maximum. This is caused by mixing phenomena throughout the extraction zone; it has also been observed by Vermijs and Kramers.[2]

(2) The efficiency increases with increasing diameter of the rotor discs.

(3) The efficiency decreases more or less rapidly with increasing diameter of the opening in the stator discs.

(4) For small heights of the compartments the number of stages per metre of contactor

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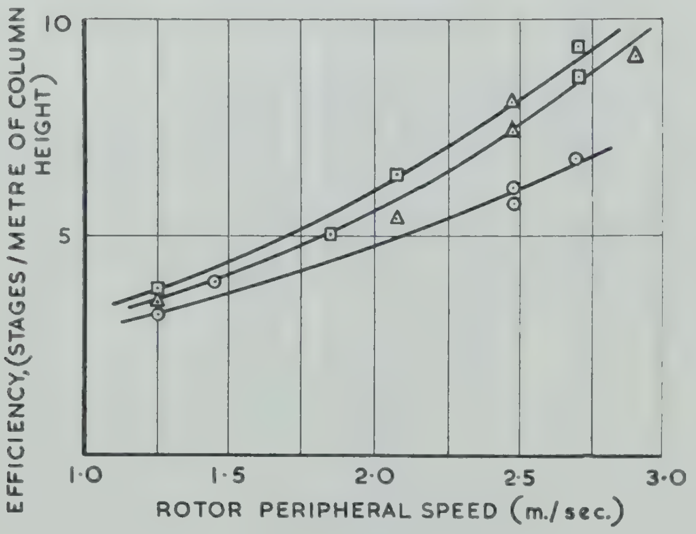
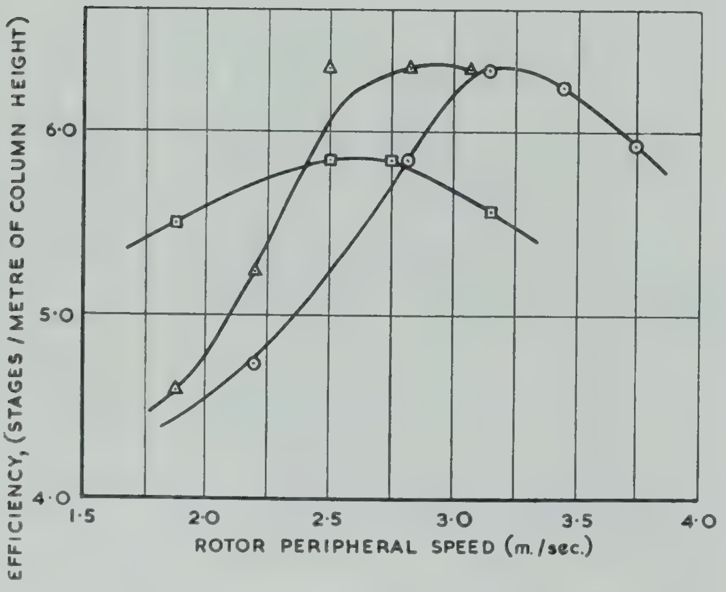
* *Viz.* (1) water–methyl-*iso*-butyl ketone (M.I.B.K.)–acetic acid and (2) water–kerosine–*n*-butylamine.



□ $V_d + V_c = 11{\cdot}2 \times 10^{-3}$ cu. m./sq. m. sec.
△ $V_d + V_c = 8{\cdot}0 \times 10^{-3}$,, ,,
○ $V_d + V_c = 5{\cdot}0 \times 10^{-3}$,, ,,
$V_d/V_c = 1{\cdot}0$

System; M.I.B.K.—Water—Acetic acid.
Water as dispersed phase.
200 mm. rotating disc contactor.
Rotor diam., 80 mm.
Stator opening, 125 mm.
Compartment height, 40 mm.
21 compartments.

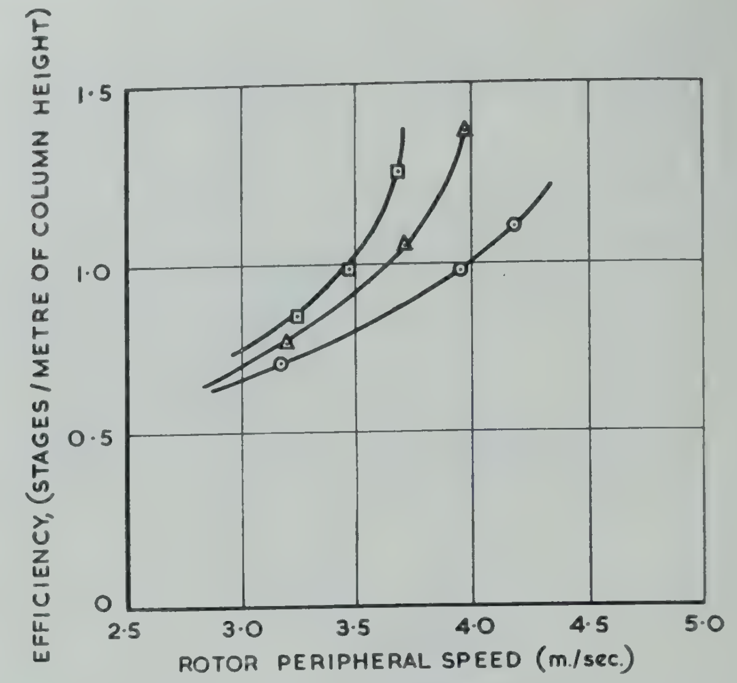
Fig. 4.—*Effect of rotor speed on efficiency for various loads*



□ $V_d + V_c = 11{\cdot}8 \times 10^{-3}$ cu. m./sq. m. sec.
△ $V_d + V_c = 8{\cdot}0 \times 10^{-3}$,, ,,
○ $V_d + V_c = 5{\cdot}0 \times 10^{-3}$,, ,,
$V_d/V_c = 1{\cdot}0$

System: M.I.B.K.—Water—Acetic acid.
Water as dispersed phase.
200 mm. rotating disc contactor.
Rotor diam., 120 mm.
Stator opening, 160 mm.
Compartment height, 60 mm.
14 compartments.

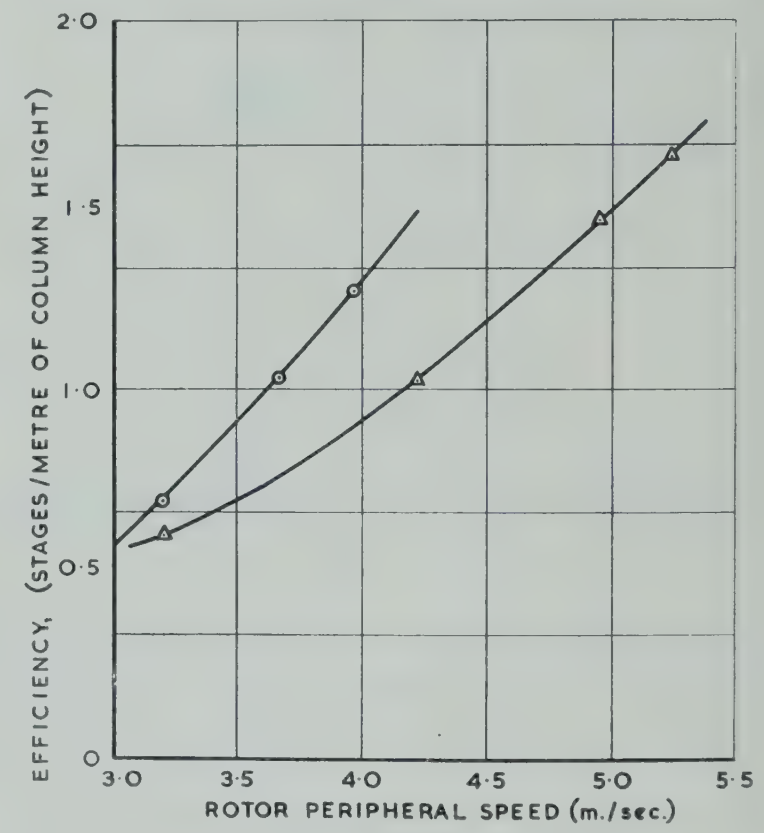
Fig. 5.—*Effect of rotor speed and specific load on efficiency (Mixing throughout the extraction zone is effective)*



Rotor diam.
○ = 200 mm.
△ = 250 ,,
□ = 290 ,,

System: Kerosene—Water—*n*-butylamine.
Kerosene as dispersed phase.
400 mm. rotating disc contactor.
Stator opening, 300 mm.
Compartment height, 100 mm.
12 compartments.
Water rate $0{\cdot}24 \times 10^{-3}$ cu. m./sec.
Kerosene rate, $0{\cdot}76 \times 10^{-3}$ cu. m./sec.

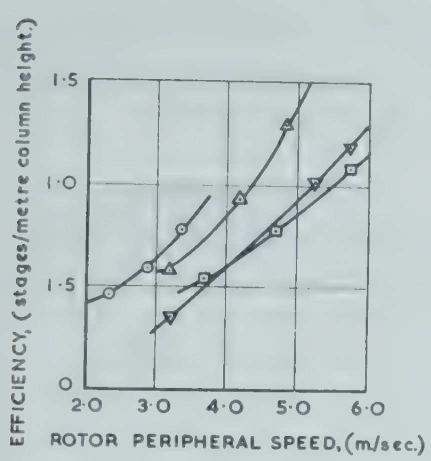
Fig. 6.—*Effect of rotor size on efficiency*



Stator
opening
○ = 260 mm.
△ = 300 ,,

System: Kerosene—Water—*n*-butylamine.
Water as dispersed phase.
400 mm. rotating disc contactor.
Rotor diam., 250 mm.
Compartment height, 100 mm.
12 compartments.
Water rate, $0{\cdot}24 \times 10^{-3}$ cu. m./sec.
Kerosene rate, $0{\cdot}76 \times 10^{-3}$ cu. m./sec.

Fig. 7.—*Effect of stator size on efficiency*



	Compartment spacing (mm.)	Contact zone (mm.)
○	60	1200
△	100	1200
□	150	1200
▽	200	1200

System: Kerosene—Water—*n*-butylamine.
Water as dispersed phase.
400 mm. rotating disc contactor.
Rotor diam. 200 mm.
Stator opening, 300 mm.
Water rate, 0·48 × 10^{-3} cu. m./sec.
Kerosene rate, 0·76 × 10^{-3} cu. m./sec.

Fig. 8.—*Effect of compartment spacing on efficiency*

height is practically constant, but it decreases with increasing compartment heights.

(5) The diameter of the shell has little influence on the efficiency.

(6) In general the efficiency increases somewhat with increasing specific load.

(7) An increase in the flow rate of the dispersed phase at constant flow rate of the continuous phase leads to a higher efficiency.

The above phenomena appear at first sight to be complicated but several of these points become clear if the contactor is considered to be equivalent to a number of ideal mixers in series through which the phases pass in counter-current flow. For such a system it can be shown (provided no entrainment of phases occurs) that the compartment efficiency η can be described accurately by the formula

$$\eta \,.\approx k\frac{a}{v}\,.\,\tau_D\,.\,E^{0.5}\text{ for }\eta \leqslant 0{\cdot}3 \qquad . \quad (2)$$

If appropriate quantities typical for the rotating disc contactor are introduced into this formula we obtain the expression:

$$\frac{\eta}{H}\approx\frac{6k}{d}\cdot\frac{h_D}{v_D}\,.\,E^{0.5}\text{ for }\eta\leqslant 0{\cdot}3 \qquad . \quad (3)$$

The limitation of compartment efficiencies to about 0·3 or less is quite realistic. Although compartment efficiencies higher than this figure can easily be obtained by increasing H, this would in fact cause a loss in separating power, since the quantity η/H, *i.e.* the number of stages per metre of contactor height, starts to drop very rapidly for values of η greater than 0·3. Furthermore, attempts to obtain high compartment efficiencies by increasing the rotor speed are in general not very successful. This is because mixing throughout the extraction zone, which affects efficiency detrimentally, then becomes fairly pronounced.

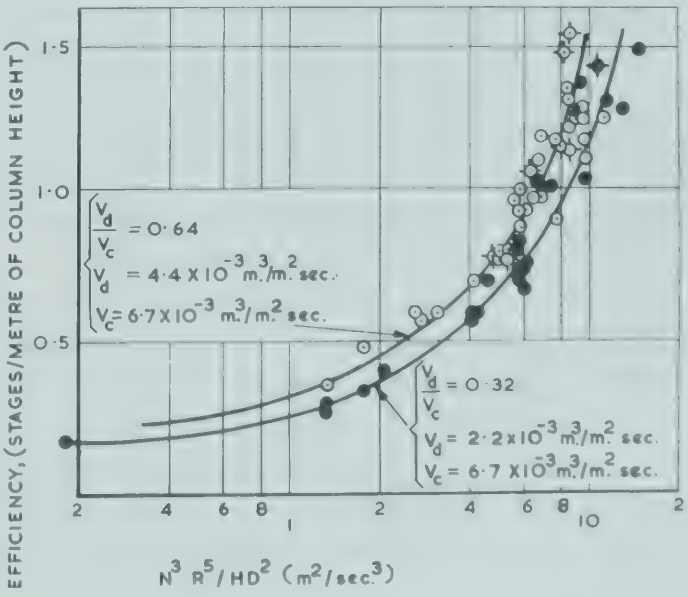
Equation (3) indicates that the quantity η/H, which is the inverse of H.E.T.S. will be suitable for correlation purposes. The drop size, d (and thus for given flow rates also the fractional hold-up of dispersed phase, h_D), will be determined by the energy input, *i.e.* N^3R^5/HD^2.

The mass transfer coefficient, k, may furthermore also be assumed to be determined by the energy input. Experiments on mass transfer in stirred vessels support this view.[3, 4]

Hence it appears that η/H and N^3R^5/HD^2 are the main variables for the correlation of efficiency data, provided mixing throughout the extraction zone can be neglected. The success obtained in using these variables may be judged from Fig. 9.

The effect of the flow rate of the dispersed phase on the efficiency can easily be understood. Because of the hindered settling of the drops, the hold-up of dispersed phase increases more than the flow rate of the dispersed phase. Accordingly the efficiency will then increase. The same holds true for the influence on efficiency of the total flow rate at constant phase ratio.

For the case illustrated in Fig. 9, the efficiency is independent of column diameter—at least up



-○- -●- 100 mm. contactor
○ ● 400 ,, ,,
(Various S.R.H.)
System: Water—Kerosene—Butylamine
Water as dispersed phase.

Fig. 9.—*Efficiency for system Water—Kerosene—Butylamine*

to column diameters not larger than 0·4 m.—in accordance with the equation given. In general one may expect that the efficiency is nearly or wholly independent of column diameter, provided mixing throughout the extraction zone is small. It is difficult to obtain confirmation of this for column diameters up to 2 or 3 m. However, results obtained in the extraction of lubricating oils with furfural (a case where mixing throughout the extraction zone is negligible) indicate the above to be correct. In this extraction, the H.E.T.S. is practically independent of energy input for energy inputs lower than those given by $N^3R^5/HD^2 = 0{\cdot}1$ (practical operating region for this case). Efficiency data obtained for this range of energy inputs with three contactors are given in Table I.

TABLE I.—*Efficiency Data for Three Rotating Disc Contactors*

Dimensions of contactor			
Diameter (mm.)	Height (m.)	Total number of theoretical stages	H.E.T.S. (m.)
64	3·0	6·5	0·45
640	3·6	6	0·60
2000	5·0	⩾7*	0·70

* Pinch conditions prevent a more accurate determination of the number of theoretical stages.

These data indicate a slower decrease in the efficiency η/H with column diameter (from 0·06 to 2·0 m.) than shown by the formula $\eta/H \approx D^{-0.15}$.

In general the scaling-up of efficiency can safely be done with the formula $\eta/H \approx D^{-0.15}$ for energy inputs lower than those given by $N^3R^5/HD^2 = 0{\cdot}1$. For energy inputs of this magnitude, the effect of mixing on efficiency can in most cases still be neglected. The situation changes when higher energy inputs are involved. The effect of mixing can no longer be disregarded, and becomes important in scaling-up. A more rapid decrease of efficiency with column diameter is observed. No general rules can be given.

The whole problem of the effect of mixing must be treated more fundamentally. The solution involves the mathematical treatment of the effect of entrainment in counter-current extraction systems where an extraction equilibrium is not reached in the stages, and also the determination and correlation of mixing throughout the extraction zone of rotating disc contactors. Both problems are still under investigation, and a discussion at this moment is premature.

Acknowledgment

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Symbols Used

a/v = surface/volume ratio of dispersed phase.
C_R = factor accounting for restriction of column by the stator rings and rotor discs in capacity correlation: equal to either S^2/D^2 or $(D^2 - R^2)/D^2$.
D = diameter of column (m).
d = droplet diameter.
E = extraction factor equals (distribution coefficient of solute) × (flow rate of solvent phase)/(flow rate of raffinate phase).
H = compartment height (m).
h_D = fractional hold-up of dispersed phase.
k = mass transfer coefficient.
N = rotor speed (rev./sec.).
P = energy input per rotor disc of contactor.
R = diameter of rotor disc (m.).
S = diameter of opening in stator disc (m.).
v_C = superficial velocity of continuous phase (m./sec.).
v_D = superficial velocity of dispersed phase (m./sec.).
η = compartment efficiency, number of theoretical stages.
ρ = density of liquid phase.
τ_D = residence time of dispersed phase in compartments (sec.).

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PROBLEMS ENCOUNTERED IN STIRRED SLURRY REACTORS

By Dr. Ir. J. G. van de VUSSE*

SUMMARY

In processes carried out in a liquid and a solid phase, the type of the reactor and the conditions of stirring in the reactor can have a far-reaching effect on the conversion and the rate of transfer of the process concerned.

Results from small-scale model tests, together with reliable scaling-up rules, will give the required data for the design of large-scale reactors. The present communication discusses the aspects of slurry reactor operation listed below, and it deals with the rules for scaling-up in each case:

1. **conditions under which all particles are just suspended,**
2. **rate of heat and mass transfer,**
3. **prevention of clogging of filters inside the reactor in cases where the solid has to be retained in the vessel in a continuously-operating process, and**
4. **conditions of immediate mixing of feed streams with reactor contents in order to prevent the development of side reactions.**

Introduction

Slurry reactors are used for processes involving a liquid-solid system in which the liquid is the continuous phase and the solid the dispersed phase. If stirring is carried out in such a reactor, the consequent mixing promotes the interchange of volume elements of the liquid and of solid particles, with the result that the transfer of mass, heat, or momentum or any combination of these (in the liquid and between liquid and solid phases) may be improved. This result is important in many physical and chemical processes.

The degree of mixing and the rate of transfer will depend upon the turbulence created by the stirrer and on the properties of liquid and solid. How, and to what extent, the transfer may be improved will depend on the requirements of the process. An analysis of the latter with the object of finding its rate-determining factors will therefore be of value for indicating the most suitable type of stirred reactor for the case in point.

For the ultimate design of reactor and stirrer, not only must the rate-determining factors be known, but also the quantitative relationship between these factors and the stirrer variables.

As will be shown later, such a relationship is only known in a limited number of cases, so that one frequently has to find a solution by means of experiments. Since such experiments are generally carried out on a small scale, the appropriate scaling-up rules require to be known for designing equipment of commercial size.

These scaling-up rules will generally differ for each individual problem. For an adequate discussion of scaling-up in slurry reactors, the various problems encountered with this type of reactor will be discussed first. Then, after a general discussion of scaling-up rules, the more specific scaling-up problems will be dealt with in more detail.

Problems Encountered in Design

Processes, in which liquid-solid systems are involved, can be divided into (*a*) physical processes, *e.g.* crystallisation, solution, melting, solidification, adsorption, extraction, flocculation, and (*b*) chemical processes, *e.g.* catalytic conversion processes, conversion processes combined with a physical process (for instance polymerisation combined with flocculation).

In general, the first requirement in such systems is that all the solid particles shall be in suspension, in other words, the state of stirring at which all particles are just suspended is of primary importance. A discussion of the scaling-up of this condition will therefore be given.

In the physical slurry-processes, mass transfer rates and heat transfer rates further dictate the efficiency of mixing, whilst for chemical processes these rates may also become the controlling factor in the overall reaction rates observed. This subject has been investigated extensively; the

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resulting scaling-up rules for heat and mass transfer rates are also included in the discussion. In catalytic reactors the reaction liquid has often to be removed by filtration, the catalyst being retained in the reactor. Stirring conditions then have to be adjusted so that no clogging of the filter occurs ; certain scaling-up rules for this condition can be given.

Finally, there is the interesting case where, in a chemical process, the conditions have to be so regulated that simultaneous side reactions are suppressed. This of course can only be done if the order of the main reaction and the side reaction are different. As an example, consider the following case in which the side reaction progresses according to a higher order than the main reaction :

$$A \xrightarrow{k_1} B \quad \text{main reaction}$$

$$A \xrightarrow{k_2} C \quad \text{side reaction.}$$

Both reaction rates are functions of A, *viz.* $k_1 f_1(A)$ and $k_2 f_2(A)$ respectively where $f_2(A)$ has a higher order in A than $f_1(A)$.

The amount of product converted into B will be :

$$M_1 = \int_\theta \int_V k_1 f_1(A)\, dt\, dV$$

and the amount of product converted into C :

$$M_2 = \int_\theta \int_V k_2 f_2(A)\, dt\, dV.$$

The integrations are carried out over the reaction time θ and the reactor volume V.

The conditions must be so chosen that the ratio M_2/M_1 lies below a certain value, which means that the concentration of A in the reactor must be kept low. In continuous operation the incoming stream, which has a high concentration of A, must, after entering the reactor, immediately be diluted with its contents. Thus in this case scaling-up rules for the condition of immediate mixing have to be known in order to design a commercial plant on the basis of successful pilot plant data.

Scaling-up Rules

The scaling-up rules generally give a relation between the scale of the equipment and some stirring variable. By using this relation, the magnitude of the stirring variable for the large-scale reactor can be found.

In cases where no reliable scaling-up rules are available, and where these cannot be derived from a process analysis and small-scale experiments, one of the following rules is frequently applied in stirring operations.

(*a*) the Reynolds number ($\rho n L^2/\mu$) must be kept constant,

(*b*) the Froude number ($n^2 L/g$) must be kept constant,

(*c*) power consumption of stirrer per unit of liquid volume (P/V) must be kept constant, and

(*d*) peripheral stirrer speed must be kept constant.

The first two quantities are dimensionless numbers (or similarity criteria), which can be derived either with the aid of dimensional analysis or from the Navier-Stokes equation. The quantities under (*c*) and (*d*) are dimensional and a clear physical similitude is not so obvious. It is always possible, however, to convert these quantities into dimensionless numbers by combining them with other factors, *e.g.* liquid and solid properties. As a large number of combinations is generally possible and the right one is unknown, and furthermore as liquid and solid properties will be unchanged on scaling-up, the application of these numbers in a dimensional form is justified. The rule of a constant power consumption per unit of liquid volume for scaling-up in particular, is frequently applied in stirring operations.[1]

If the small-scale experiments are carried out in regular models, geometrical similarity will exist on scaling-up. In that case the scaling-up rules mentioned above give a relation between the scale (some length ratio L) and the stirrer speed n, *viz.* :

(*a*) nL^2 is constant,

(*b*) n^2L is constant,

(*c*) $\left[\begin{array}{l} n^3L^2 \text{ is constant if } P \propto \rho n^3 L^5 \text{ (turbulent region)} \\ n \text{ is constant if } P \propto \mu n^2 L^3 \text{ (viscous region), and} \end{array}\right.$

(*d*) nL is constant.

It must be borne in mind that the application of one of these rules, without sufficient knowledge of the determining process factors involved, may lead to undesirable consequences in large-scale operation. To a certain extent this can be avoided by carrying out the process in semi-scale equipment, where the reliability of the scaling-up rule can be checked.

Conditions under which all particles are just suspended

The conditions required for just maintaining all particles in suspension will be discussed for two types of reactor:

(*a*) reactors in which agitation is such that an isotropic turbulent field is created by the stirrer, and

(*b*) reactors in which a non-isotropic turbulent field is present during stirring.

(*a*) ISOTROPIC TURBULENCE

As a rule the turbulent field which is created by a rotating stirrer will be non-isotropic: regions with high intensities of turbulence exist in the neighbourhood of the stirrer, whereas regions with lower intensities occur in the corners of the reactor. If, however, a reactor can be equipped with a large number of small stirrers, the overall turbulent field may be considered as isotropic, although the field around each stirrer will be non-isotropic. An example is the stirred tube reactor, where an axial rotating shaft is equipped with a number of small stirrers. In this reactor isotropy of the turbulent field exists in an axial direction.

An important aspect of the stirred tube reactor is the possibility of "staging" it; this possibility may offer particular advantages for certain types of processes. For an isotropic turbulent field, a mathematical description of the hydrodynamic behaviour of the liquid-solid system and a mathematical derivation for the condition under which all particles are just suspended can be given.

In isotropic turbulence the concept of an eddy diffusivity can be used. The eddy diffusion coefficient E is related to the scale (l) and the intensity (v) of turbulence in such a way that $E \propto lv$.

The concept of an eddy diffusion applies both to the liquid and the solid phases. The diffusion of solid particles suspended in a fluid in turbulent motion is very similar to the behaviour of colloidal particles in Brownian motion. A fundamental study of this subject has been given by Tchen Chan Mou[8] and by Loo.[6] The latter author shows by theoretical deduction that the eddy diffusivity of the particles is greater than that of the liquid, but tends to that of the liquid for small particles. Generally both diffusivities will be equal if the particle diameter is small compared with the scale of turbulence. As the scale of turbulence in a stirred tube reactor is of the order of magnitude of the stirrer diameter, the particle diameter will in most cases be small relative to the scale of turbulence, so that the diffusivities of the liquid and solid phases may be taken to be equal.

Application of the diffusion equation for the particles suspended in a vertical stirred tube reactor gives, in axial direction:

$$E\frac{dc}{dz} - vc = \text{constant} = -q \qquad (1)$$

where

E = axial eddy diffusion coefficient,
c = concentration of particles,
v = particle velocity (settling velocity minus fluid velocity),
q = current of particles,
z = axial co-ordinate.

If no particles are introduced into the field or are removed from it, q must be zero. Hence

$$E\frac{dc}{dz} - vc = 0$$

and

$$\ln\frac{c}{c_0} = \frac{vz}{E}, \qquad (2)$$

where c_0 = concentration of particles at the top of the reactor where $z = 0$.

The average concentration of particles can be found from:

$$\bar{c} = \frac{1}{Z}\int_0^Z c\,dz = \frac{Ec_0}{vZ}\left(e^{\frac{vZ}{E}} - 1\right), \qquad (3)$$

where Z = depth of liquid in reactor.

If ϕ is written for the dimensionless quantity vZ/E, we get

$$\frac{\bar{c}}{c_0} = \frac{1}{\phi}\left(e^{\phi} - 1\right). \qquad (3a)$$

The average concentration $\bar{c}$ must be equal to the total amount of solid particles divided by the volume of liquid in the reactor.

The conditions under which all particles are just in suspension can be found by the following reasoning. Under conditions of rest with a motionless stirrer, a bed of particles will be on the bottom of the reactor. If Z_1 is the depth of the particle bed and c^* is the concentration of particles in the bed, then

$$Z\bar{c} = Z_1 c^*.$$

If stirring is now started, some of the particles will go into suspension; their distribution along the column will be a logarithmic one according to equation (2), the concentration rising with depth until it reaches c^* at the point where the bed begins. With increasing speeds more particles will be suspended and the depth of the particle bed at the bottom will diminish. At a certain speed the particle concentration at the bottom ($z = Z$) is still equal to c^* but $c < c^*$ at $z \leqslant Z$.

A further increase of the stirrer speed would give a concentration at the bottom which is lower than c^*, ($c < c^*$ at $z = Z$).

This signifies that all particles are suspended, if $c \leqslant c^*$ at $z = Z$ and $c < c^*$ at $z < Z$. This result leads to:

$$\frac{\phi e^{\phi}}{e^{\phi}-1} \leqslant \frac{Z}{Z_1} \quad . \quad . \quad . \quad (4)$$

In most practical cases $\phi > 1$ and then

$$\frac{e^{\phi}}{e^{\phi}-1} \approx 1,$$

so that equation (4) can be simplified to

$$\phi \leqslant \frac{Z}{Z_1} \text{ or } E \geqslant vZ_1 \quad . \quad . \quad (5)$$

Expressed in words: All particles are just maintained in suspension if the eddy diffusion coefficient is just equal to the product of the particle velocity and the height of the particle bed that would exist if all particles were on the bottom of the reactor.

The rules for scaling-up can be derived from equation (5) and an expression for the eddy diffusivity in terms of stirrer variables. As has been stated before, the eddy diffusivity is proportional to the product of the scale and the intensity of turbulence. Now in the turbulent region, the scale of turbulence will be proportional to a stirrer dimension (L) whilst the intensity will be proportional to the stirrer speed (nL). Hence in the turbulent region $E \propto nL^2$. This means that the dimensionless group nL^2/vZ_1 must be kept constant when scaling-up.

If the particle velocity v remains the same and if the bed height Z_1 varies in proportion to the scale ($Z_1 \propto L$), we find that the peripheral rotor speed must be kept the same.

In the viscous region the group E/nL^2 will be a function of the Reynolds number ($\rho nL^2/\mu$), in such a way that at lower Reynolds numbers E/nL^2 will decrease. Since in scaling-up the Reynolds number will increase at equal peripheral speeds, a lower value for nL^2/vZ_1 may be used in the large-scale reactor than would follow from the small-scale experiments.

If equal values for nL^2/vZ_1 are taken, there will always be a margin of safety in scaling-up.

(b) NON-ISOTROPIC TURBULENCE

In reactors or mixing vessels where only one stirrer or a small number of stirrers is present, the turbulent field will be non-isotropic.

The concept of an eddy diffusivity can no longer be applied and only a phenomenological description of the process of suspending the particles can be given. Experiments in stirred slurry reactors were carried out by Hirsekorn and Miller[2] for the viscous region and by Kneule[5] for the turbulent region.

Observations made during these tests showed the following stages of progress in the hydrodynamic behaviour of the suspension as the agitator speed is increased incrementally. If the agitator is rotated slowly, a mound of particles is first formed directly beneath the shaft, the particles flowing out towards the tip of the paddle and then returning beneath the shaft. This gives the appearance of a torus under the paddle. With an increase in paddle speed some of the solids leave the torus and rise along the wall of the vessel to a level which is determined by the flow conditions of the liquid and by the settling velocity of the solid particles. These particles then move in towards the shaft and down to the paddle. More particles follow the same path until a steady state is reached where the number of particles following this route above the paddle is constant. A further increase in speed reduces the number of particles at the bottom still further and more particles are suspended.

Finally a state will be reached at which all particles are just in suspension. The minimum rotor speed pertaining to this state has been investigated extensively as a function of characteristic variables.

Kneule and Bogenstätter[5] relate the condition in which all particles are just maintained in suspension to the power consumption of the stirrer. They state that in order to maintain all particles in suspension a power consumption is needed which is equal to the product of the particle settling velocity v and the submerged weight of the particles $V_d\,(\rho_d - \rho_c)\,g$. Furthermore, a certain power consumption is required for the amount of liquid to be moved. The following relation was found[5]:

$$P \propto vg\,(\rho_d - \rho_c)\,V_d \times \left(\frac{V_c}{V_d}\right)^n$$

The exponent n appears to be $0 \cdot 5$ for $\frac{V_c}{V_d} < 2000$. Hence in the turbulent region the following relation can be used :

$$P = C_1 (W_d W_c)^{0.5} \, v \, \frac{\Delta\rho}{(\rho_d \rho_c)^{0.5}} \quad . \qquad (6)$$

The constant C_1 is dependent on the type of stirrer employed. It appeared that paddle stirrers need a 4- to 5-fold higher power consumption than propeller stirrers. The reciprocal value of C_1 is a measure of the efficiency of the stirrer as a means for suspending particles.

For scaling-up purposes, it is useful to express P in terms of stirrer variables. Appropriate correlations can be found in literature.[7] In the case, for instance, of a fully baffled stirred reactor $P \propto \rho_c n^3 L^5$. In this case the rule for geometrical scaling-up will be that $n^3 L^2$ must be kept constant.

In the viscous region the dimensionless group

$$C_1 = \frac{P}{v \Delta\rho g (V_d V_c)^{0.5}}$$

will be a function of the Reynolds number $\rho n L^2/\mu$, in such a way that C_1 increases at lower values of the Reynolds number. This may be due to the fact that in the viscous region a much greater part of the stirrer energy will be consumed by the viscous liquid flow, so that a lower "efficiency" of the stirrer results.

In the viscous region $P \propto \mu n^2 L^3$. If the power input per unit of liquid volume were again kept constant in scaling up, this would imply that n remains constant and the Reynolds number increases in proportion to the square of the scale factor.

As, however, C_1 decreases at higher Reynolds numbers, lower power inputs per unit of liquid volume may be applied in the large-scale reactor than were found from the model experiments. Hence the rule of equal power input per unit of liquid volume will lead to a safe design.

Rate of mass transfer and heat transfer

Hixson and Baum[3, 4] extensively investigated the solution and melting of particles in a stirred reactor. Mass transfer rates from the dissolving particles into the liquid and heat transfer rates from the liquid into the melting particles were investigated as a function of stirring variables for various liquid-solid systems. It was found that in both systems the transfer rate rapidly increases with stirrer speed as long as the particles are not completely suspended. As soon as the state of complete suspension is reached, the transfer rate increases but slowly with stirrer speed. This conclusion has been confirmed by Kneule[5] who considered the state of complete suspension as an optimum condition.

For the mass and heat transfer rate under the condition in which all particles are just suspended, the following expressions are valid :[3, 4, 5]

Mass transfer :

$$\frac{K}{(2g\nu)^{1/3}} = 9 \times 10^{-2} \left(\frac{D}{\nu}\right)^{0.5} \left(\frac{\Delta\rho}{\rho_c}\right)^{1/3}$$

Heat transfer :

$$\frac{h}{C_p \rho (2g\nu)^{1/3}} = 11 \cdot 5 \times 10^{-2} \left(\frac{k}{\mu C_p}\right)^{0.5} \left(\frac{\Delta\rho}{\rho_c}\right)^{1/3}$$

In these equations reactor and stirrer dimensions do not occur. It follows that if, in scaling-up the conditions set forth in the previous paragraph are satisfied, scaling-up of the rates of heat and mass transfer will be obtained automatically.

Prevention of clogging of filters in stirred reactors

If, in a catalytic process, the catalyst is retained inside the reactor whilst the reaction liquid is removed continuously by filtration, clogging of the filter may occur. At high filtration rates this may be very troublesome.

In order to prevent the building-up of filter cake, stirring can be applied. The higher the stirrer speed, the higher can be the filtration velocity before clogging occurs. The condition under which all particles are just kept in suspension will depend on the dimensionless ratio of the peripheral stirrer speed and the filtration velocity. This ratio must lie above a critical value, which can be determined from model experiments.

It may be expected that in scaling-up no clogging will occur if this ratio is always kept above the critical value.

Condition of immediate mixing

For preventing high–order side reactions, the feed introduced into a reactor has to be mixed immediately with the liquid contents of the reactor. The introduction of the feed in the neighbourhood of the stirrer will improve the rapid distribution through the liquid. In small-scale experiments the stirring necessary to fulfil the mixing requirements, *i.e.*, to suppress the side reaction rate below an acceptable level can be determined. Equivalent results may then be expected on scaling-up, if under conditions of equal residence times, the time necessary to dilute the incoming stream with the reactor contents can be kept as low, in the large-scale reactor, as it was in the small-scale reactor. This time is closely related to the mixing time and will be unchanged if the mixing time and the turbulence is kept the same in upscaling.

Expressions for the mixing time in terms of stirrer variables can be found in literature.[9] In these expressions the mixing time is related to the pumping capacity of the stirrer. The mixing time appears to be approximately proportional to the time required for circulating the liquid once, this time being equal to the ratio of the liquid volume to the pumping capacity of the stirrer. Roughly speaking, the pumping capacity of the stirrer is proportional to nL^3. On scaling-up the volume of the reactor will be proportional to L^3. Hence the mixing time will remain unchanged if the number of revolutions of the stirrer is the same in the large and small apparatus.

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Symbols Used

c = concentration (kg. mol. m.$^{-3}$).
C_p = specific heat at constant pressure (J. kg.$^{-1}$ °C.$^{-1}$).
D = diffusion coefficient (m.^{2} sec.$^{-1}$).
E = eddy diffusion coefficient (m.^{2} sec.$^{-1}$).
g = acceleration of gravity (m. sec.$^{-2}$).
h = heat transfer rate (J. sec.$^{-1}$ m.$^{-2}$ °C.$^{-1}$).
k = thermal conductivity (J. sec.$^{-1}$ m.$^{-1}$ °C.$^{-1}$).
K = mass transfer rate (m. sec.$^{-1}$).
l = length (m.).
L = length of stirrer (m.).
M = mass (kg. mol.).
n = number of revolutions of stirrer (sec.$^{-1}$).
P = power consumption (w.).
q = flow rate (kg. mol. m.$^{-2}$ sec.$^{-1}$).
t = time (sec.).
v = velocity (m. sec.$^{-1}$).
V = volume (m.^{3}).
W = weight (N.).
z = vertical co-ordinate (m.).
Z = height, depth (m.).
μ = dynamic viscosity (kg. m.$^{-1}$ sec.$^{-1}$).
ν = kinematic viscosity (m.^{2} sec.$^{-1}$).
ρ = density (kg. m.$^{-3}$).
ϕ = dimensionless quantity $\left(\frac{vZ}{E}\right)$.

Subscripts

$_c$ = continuous phase.
$_d$ = dispersed phase.

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MODEL TESTS WITH CYCLONES

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SUMMARY

An investigation has been made to find out whether information on an industrial cyclone may be obtained from model tests or from full-scale experiments carried out under conditions that cannot be realised in the model.

It appears from this study that it is frequently impossible to satisfy the model laws, which is a condition necessary for obtaining the data needed. However, the necessary information may be derived from calculations. The method of calculation is set down and explained in this paper.

Introduction

Cyclones for blast furnaces may have a handling capacity of 100 000 cu. metres/hr. and upwards at N.T.P. They are capable of separating about 1·5 tons of dust per hour.[1] The gas-dust mixture has an average temperature of 250° C. and is subjected to a pressure of about 300 kg./sq. metre gauge. The pressure drop across the cyclone is taken to be almost 100 kg./sq. metre. Such a cyclone measures 20–25 m. in height and has a cylindrical section of 5–6 m. diam.

It will be clear that this large energy consumption and the high capital charge, make it desirable to estimate the expected costs.

Every dust separator has to satisfy the following requirements[2]

(*a*) good separating performance,
(*b*) small pressure loss,
(*c*) small overall dimensions,
(*d*) low maintenance costs, and
(*e*) low price.

The cyclone dust separator has a low price and is cheap to maintain. If the other three conditions are also to be fulfilled, it will be necessary, by carrying out experiments or otherwise, to form an idea of the probable energy consumption, separating performance, and dimensions. The large volumes handled and the working conditions often make it impossible to carry out these experiments on an industrial scale. Consequently, one has to depend on smaller-scale tests, in some cases even using model gas-dust mixtures. However, if these tests are to be of any value for the design of the ultimate plant it is necessary to know something about the model laws holding for cyclones.

These model laws are concerned not only with the scale of operation but also with the type of model substances used.

Example

By way of example, consider the case of well-defined fine crystals which have to be separated in a cyclone from a valueless suspension of by-products. Such a test may often be carried out on a full scale, although not with the actual substances because of the large capacity of the cyclone. Moreover handling the quantities available for test would require the use of a cyclone of impossibly small dimensions. A 50 mm. cyclone for separating out crystals of 100 μ has a throughput of 4 cu. metres/hr. at N.T.P.

The necessary data will therefore have to be derived from experiments using a model substance and a cyclone of true dimensions. Here again, some knowledge of the model laws is required.

Operation of the Cyclone

Dust-laden gas enters the cyclone through a tangential feed channel and flows along helical paths towards the axis where it is discharged through two or more apertures. This helical flow may be considered to be composed of a rotary movement superposed on a radial flow towards the axis. Because of the rotary flow, the solid particles in the cyclone are subjected to a centrifugal force directed contrary to the force exerted on the particles by the axial flow. The separating effect of the cyclone depends on the ratio between these two forces.[3] Particles of a sufficiently high settling rate are driven towards the outer wall of the cyclone by the action of the centrifugal force and carried towards the apex aperture through a boundary layer along the wall.[4]

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Particles of lower settling rates are entrained by the axial gas flow and issue through the central overflow aperture. It may be assumed[5] that the separation between particles that are caught and particles that are entrained by the gas flow takes place on a cylinder surface the diameter of which equals that of the overflow aperture. The size of the smallest particle that may still be caught (the equilibrium grain) will then depend on :

(*a*) the operating conditions, such as the ratio between the specific weight of the particles and the carrier fluid, the viscosity of the gas, etc., and

(*b*) the geometric design of the cyclone.

The fundamental factors to be considered in designing a given cyclone are its pressure drop and separating performance. Both factors will be a function of the flow pattern and the centrifugal forces. At equal Reynolds numbers cyclones of the same geometric design show the same flow pattern: at equal Froude numbers they show the same ratio between centrifugal and gravity acceleration. The separating effect will depend on the ratio of the specific gravities of the particles and of the carrying fluid. In making a comparison between model and reality, the following three dimensionless coefficients have to be considered :

(*a*) the flow factor, which is a function of the Reynolds number,

(*b*) a centrifugal force coefficient,[1] which may be identified with the Froude number, and

(*c*) a coefficient which is a measure of the separating performance of the cyclone.

Model Cyclone

Tests have been made with a cyclone of the same type as described by ter Linden.[6] In this model part of the feed channel wall is extended round half the circumference of the cylindrical section. As a result, the central vortex is disturbed as little as possible by the entering gas.

The dimensions of this cyclone are :

$F_0 = a_0 \times b_0 = 30 \times 16 = 480$ sq. mm.
$D_1 = 100$ mm.
$F_1 = 7854$ sq. mm.
$D_2 = 27$ mm.
$F_2 = 573$ sq. mm.
$D_3 = 15$ mm.
$F_3 = 177$ sq. mm.
$\gamma = 10°$,
$h_1 = 50$ mm.
$h_2 = 35$ mm.
$H = 290$ mm.

The quantity and the temperature of the inlet gas (air) were measured and the difference between feed pressure and atmospheric pressure was determined. The overflow aperture opened freely into the atmosphere and the apex aperture into an air-tight collecting chamber. The volume of air was varied from 13 cu. m./hr. to 34 cu. m./hr. and the pressure from 30 kg/sq.m.g. to 300 kg./sq.m.g. In this range, particles of over 10–15 μ and a specific weight ratio of 2×10^3 are separated out.

Industrial Cyclone

Imagine that it is desired to know the dimensions and the probable pressure drop for the cyclone mentioned at the beginning of the introduction. Imagine that about 200 000 cu.m./hr. of a gas, density $0 \cdot 070$ kg.sec.^{2}/m.^{2} and a kinematic viscosity of $42 \cdot 10^{-6}$ m.^{2}/sec., have to be handled and that particles of over 50 μ have to be separated from the gas. The specific weight ratio is $2 \cdot 10^3$.

To obtain an idea of the cyclone dimensions we shall assume that the cyclone is identical to the model cyclone and select a feed rate. In making this choice we are limited by the compressibility of the gas. At a feed rate of only 20 m./sec. the compressibility effect will be noted in the core and give rise to an energy loss. Taking 20 m./sec. as the limit rate, the dimensions of the cyclone calculated from these data are as follows :

$F_0 = 2 \cdot 28 \times 1 \cdot 22 = 2 \cdot 78$ sq. m.
$D_1 = 7 \cdot 61$ m.
$D_2 = 2 \cdot 06$ m.
$D_3 = 1 \cdot 14$ m.
$\gamma = 10°$.
$h_1 = 3 \cdot 81$ m.
$h_2 = 2 \cdot 66$ m.
$H = 22 \cdot 10$ m.

To enable this cyclone to be related to the model cyclone, we shall define the Reynolds number for a cyclone by

$$Re = \frac{V_0 D_1}{\nu}$$

and the Froude number by

$$Fr = \frac{V_0^2}{D_1 g}.$$

We thus find that, whereas for the model $Re^* = 1{\cdot}3 \times 10^5$ and $Fr^* = 407$, the corresponding values for the designed cyclone are

$$Re = 3{\cdot}6 \times 10^6$$

and $Fr = 5236$. From this it follows that the designed cyclone will give a separating performance considerably poorer than that of the model. No prediction can be made, however, as to the pressure loss (which for the model amounted to 310 kg./sq. m.), because the flow pattern will differ greatly from that in the model cyclone owing to the large difference between the Reynolds numbers.

The Reynolds and Froude numbers of the model may be more in line with those of the designed cyclone by giving the model a diameter of 50 cm. and operating it with water. However, this requires an enormous amount of water to be pumped notwithstanding the inlet velocity being 5·13 m./sec. In addition, it should be seen too that the pressure in the system will nowhere drop below the vapour pressure of the water. Furthermore, the third model law, saying that the ratio of specific gravities should remain constant, cannot be satisfied. Under these conditions, therefore, the model can only furnish information on the pressure and not on the separating performance.

Design of a Cyclone

In many cases the model laws do not enable a proper choice to be made of the design of a cyclone, nor of its properties. However, by using Taylor's calculation[4] for the thickness of the boundary layer, some further data may be computed.[7]

Defining a flow factor α by

$$Q = \alpha F_0 \sqrt{\frac{2\Delta P}{\rho}}$$

the probable pressure loss at a given load Q may be calculated from :

$$p_0 - p_2 = (\alpha^{-2} - 1) \cdot \tfrac{1}{2}\rho V_0^2 + \tfrac{1}{2}\rho V_2^2 \qquad (1)$$

provided α is known. For a given cyclone α may be computed from simple quantity and pressure measurements. It then appears that α is a function of the Reynolds number of the cyclone. This function may be expressed by the formula :

$$\alpha = \frac{\alpha_\infty}{1 - \frac{1}{6} G \frac{F_1}{F_0} \sqrt{\left(\frac{2}{Re \sin \gamma}\right)}} \quad . \qquad (2)$$

where α_∞ is the flow factor of a cyclone fed with an ideal fluid. $Re = \infty$ for such a case.[8] The term

$$\frac{1}{6} G \frac{F_1}{F_0} \sqrt{\frac{2}{Re \sin \gamma}}$$

is a correction for the boundary layer.

α_∞ and G are functions of D_0, D_1, and D_2 which, after calculation, may be plotted in tables or graphs. Equation (2) for α does not include a single experimental datum, but has been derived exclusively from a stylised concept of the flow pattern in the cyclone. In developing this concept it was assumed that the flow in the cyclone equals that of an ideal fluid except in the boundary layer along the wall.

When

$$\frac{1}{6} G \frac{F_1}{F_0} \sqrt{\frac{2}{Re \sin \gamma}} = 1, \ \alpha = \infty,$$

which gives a Reynolds number of

$$Re_{sp} = \frac{G^2}{18 \sin \gamma} \left(\frac{F_1}{F_0}\right)^2$$

which we shall call *the specific Reynolds number of the cyclone.**

Experiments have shown that if the cyclone is to give a good performance under the operating conditions, the Reynolds number should be an order of magnitude higher than this specific number which depends exclusively on the dimensions of the cyclone. For the assumed model cyclone for example $Re_{sp} = 1{\cdot}24 \times 10^4$, which is a very high value. For this cyclone, the choice of the ratios of the linear dimensions has not been a particularly happy one. As was stated earlier, under the operating conditions $Re^* = 1{\cdot}3 \times 10^5$.

The specific Reynolds number of a cyclone is an interesting characteristic, which enables us to find out whether a cyclone operated with a given fluid will have a reasonably good separating performance.

The physical significance of the specific Reynolds number is that the whole of the fluid that leaves the cyclone, comes from the boundary layer and is therefore not subjected to the separating forces of a vortex.

Let us imagine that the cyclone is operated with a fluid having a kinematic viscosity 100 times greater than that of water, but of the same density. In that case the model cyclone would require a feed pressure of 500 atm. to warrant satisfactory results.

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* A suggestion of Dr. F. V. A. Engel.

The usefulness of the formula for the flow factor has been tested for the model cyclone. The results of this examination are shown in Fig. 1. The pressure to be expected at various loads has been calculated using equation (1), the value of α in this formula being derived for various Reynolds number by means of equation (2). The full line gives the calculated values, measured values being indicated by circles. In these measurements the Reynolds number varied from $5 \cdot 75 \times 10^4$ to $1 \cdot 2 \times 10^5$. It appears that the greatest deviations occur at low Reynolds numbers which are only 5–6 times greater than Re_{sp}, *i.e.* in the region where these deviations may be expected because of an unduly large wall-effect.

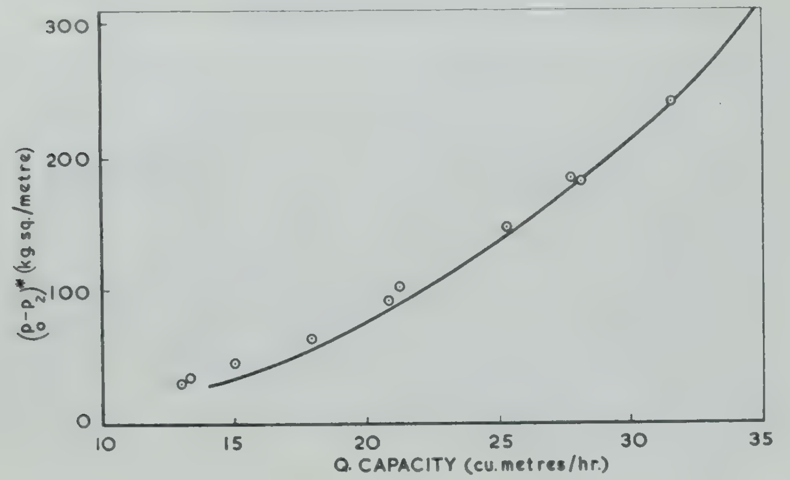

Fig. 1.—*Cyclone characteristic*

Besides the pressure, the separating performance of the cyclone also calls for attention. Assuming for simplicity's sake that the diameter of the largest particle which cannot be caught is a measure of the performance, it has been found that this diameter may also be derived by calculation only. This minimum dimension may be found using the formula :

$$d_0 = D_2 \left[0 \cdot 349 \; Re \, \frac{(D_1 - \xi D_2)}{b_0} \left(\frac{\rho'}{\rho} - 1 \right) \right]^{-\frac{1}{2}} \quad . \qquad (3)$$

where ξ is a function of D_0, D_1, and D_2. This equation which, like equation (2), does not include a single experimental value, is approximate because it has been supposed that the particles do not influence one another, and that the medium has not been loaded to such a degree that its kinematic viscosity is affected. This is also true for the calculation of the flow factor. A high load of particles results in a decrease of the pressure drop and a deterioration in performance. This may be accounted for by an increase in dynamic viscosity, thus decreasing the Reynolds number. This change in viscosity is caused mainly by particles below 5μ, which implies that if the gas contains large quantities of such particles a higher viscosity and hence a poorer performance and a lower pressure drop, are to be expected. Generally speaking, these particles cannot be caught in a cyclone. Application of equations (1), (2), and (3) to the model cyclone and the designed cyclone, give

$$\alpha^* = 0 \cdot 2795,$$
$$(p_0 - p_2)^* = 307 \text{ mm. water},$$
$$d_0^* = 5 \ \mu \text{ for the model}$$

and

$$\alpha = 0 \cdot 2052,$$
$$(p_0 - p_2) = 342 \text{ mm. water}$$
$$d_0^* = 60 \ \mu$$

for the full-scale cyclone. This means that the designed cyclone is considerably inferior to the model.

Improvement of the Design

One of the deficiencies of the model cyclone is its high specific Reynolds number

$$(Re^* = 1 \cdot 24 \times 10^4)$$

which is to be ascribed to the unfavourable shape. After making an extensive experimental study, ter Linden indicates the optimum shape.[2] According to this author the optimum ratios are :

$F_0 = F_2$,
$a_0 = b_0$,
$D_1 = 2 \cdot 25 \ D_2$,
$2\gamma = 20\text{–}30°$.

The specific Reynolds number of such a cyclone is $Re_{sp} = 905$, a much lower value than the one mentioned.

Assuming again an inlet velocity of 20 metre/sec. for this design, the principal dimensions will be :

$F_0 = F_2 = 2 \cdot 78$ sq. metre,
$a_0 = b_0 = 1 \cdot 67$ metre,
$D_2 = 1 \cdot 88$ metre,
$D_1 = 4 \cdot 25$ metre.

The Reynolds number then becomes $Re=2\times10^6$. From this value and equation (2) it follows that $\alpha = 0{\cdot}274$. Using equation (1), the pressure loss now becomes 186 mm., while the size of the equilibrium grain, calculated from equation (3), is $d_0 = 38\ \mu$.

At a lower inlet velocity of 15 metre/sec., we find a cyclone of the following dimensions :

$$a_0 = b_0 = 1{\cdot}92 \text{ metre},$$
$$D_2 = 2{\cdot}17 \text{ metre},$$
$$D_1 = 4{\cdot}90 \text{ metre},$$

giving

$$Re = 1{\cdot}75 \times 10^6,$$
$$\alpha = 0{\cdot}275,$$
$$\text{pressure loss} = 105 \text{ mm}.$$

and an equilibrium grain of 44 μ.

Using these formulae, it is possible to consider other types of cyclones theoretically. We may also start from specific requirements as to the allowable pressure drop or the equilibrium grain size or from both. In the last case it appears that it will be necessary to depend on multi-cyclones.

In practice the pressure drop will be usually somewhat lower than the theoretical value because of the presence of the solid particles, while the collecting chamber will also receive particles smaller than the equilibrium grain since the particles influence one another.

For cyclones of normal design, however, the formulae faithfully reflect the general tendencies.

Appendix

Calculation of the flow factor

As stated above :

$$\alpha = \frac{\alpha_\infty}{1 - \frac{1}{6} G \frac{F_1}{F_0} \sqrt{\left(\frac{2}{Re \sin \gamma}\right)}}$$

if

$$Re = \frac{V_0 D_1}{\nu},$$

α_∞ and G being functions of D_0, D_1, and D_2.

To determine α_∞ proceed as follows : calculate

$$\frac{F_0}{F_2} \cdot \frac{D_2}{D_1}$$

and read the corresponding value of

$$\frac{\xi^2 \sqrt{2}}{(1+\xi^2)^{\frac{1}{2}}}$$

from Table I.

TABLE I.—*Values of* $\frac{\xi^2 \sqrt{2}}{(1+\xi^2)^{\frac{1}{2}}}$

$\frac{F_0}{F_2} \cdot \frac{D_2}{D_1}$	ξ	$\frac{\xi^2 \sqrt{2}}{(1+\xi^2)^{\frac{1}{2}}}$
0·525	0·700	0·568
0·489	0·710	0·581
0·472	0·715	0·588
0·443	0·724	0·600
0·425	0·730	0·609
0·402	0·737	0·619
0·378	0·745	0·629
0·298	0·775	0·672

Using the formula

$$\frac{F_0}{F_2} \cdot \frac{D_2}{D_1} = \frac{(1-\xi^2)^{3/2}}{\xi^2 \sqrt{2}},$$

this table may be extended at will by substituting various values for ξ and calculating the first and third columns.

α_∞ is calculated by :

$$\alpha_\infty = \frac{D_2}{D_1} \cdot \frac{\xi^2 \sqrt{2}}{(1+\xi^2)^{\frac{1}{2}}}.$$

The value of G can be taken from Table II.

TABLE II.—*Values of G*

D_2/D_1	G
0·50	9·880
0·45	10·445
0·40	10·900
0·35	11·397
0·30	11·797
0·25	12·198
0·20	12·483

Table I also gives the values for ξ which occur in the formula for the equilibrium grain.

Symbols Used

a_0 = height of feed aperture.
b_0 = width of feed aperture.
d = particle diameter.
D_1 = cyclone diameter.
D_2 = diameter of overflow aperture.
D_3 = diameter of apex aperture.
F_0 = area of feed aperture.
$F_1 = \frac{1}{4}\pi D_1^2$.
F_2 = area of overflow aperture.
F_3 = area of apex aperture.
g = acceleration due to gravity.
H = overal length of the cyclone.
h_1 = height of cylindrical section.
h_2 = length of vortex finder.

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P = difference in total pressure before the cyclone and after the overflow aperture.

Q = capacity = V_0/F_0.

V_0 = inlet velocity, $V_2 = Q/F_2$.

α = flow factor.

γ = half apex angle.

ν = kinematic viscosity.

ρ = density.

An asterisk (*) refers to conditions in the model.

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THE SCALING-UP OF SOLVENT EXTRACTION PROCESSES FOR IRRADIATED NUCLEAR FUELS

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SUMMARY

The development of processes designed to recover uranium and plutonium from irradiated reactor fuel elements has involved several problems of scaling-up solvent extraction equipment. This paper describes the approach made to problems of scaling-up the packed columns and mixer-settlers used in atomic energy processes.

Introduction

Nuclear reactors of the Windscale and Calder Hall type discharge several hundred tons of irradiated uranium fuel elements per annum. These elements are stripped of their protective cans, dissolved in nitric acid, and the uranium and plutonium are separated from the highly radio-active fission product elements before being re-converted to metal for further use.

The separation and subsequent purification of the uranium and plutonium is achieved by means of a continuous, counter-current, solvent extraction process carried out in a variety of extractors. The primary separation process uses gravity-fed packed columns ; ancillary purification and recovery processes employ pulsed columns and mixer-settlers. It will be appreciated that, at the commencement of the atomic energy project in this country, it was necessary to design and erect the chemical plant simultaneously with the first reactors it had to serve. The usual course of building a pilot plant to study process variables could not be followed because there was insufficient time to build plant needed for handling highly radio-active materials and because the irradiated fuel elements could not be made available in time.

The process and plant development therefore took the course of determining the extraction chemistry of uranium, plutonium, and fission products on the scale of a few millilitres of solution and then, by using un-irradiated uranium as a key component, of determining the size and nature of the extraction equipment required.

In the present paper it is proposed to discuss the scaling-up of packed columns and mixer-settlers and to describe the approach that was used in designing plant at Windscale and elsewhere. This more empirical approach will be related to the fundamental work which has been carried out subsequently at A.E.R.E. and which has demonstrated the complexity of the problem.

Work on packed columns and mixer-settlers has been undertaken for the United Kingdom Atomic Energy Authority by Imperial Chemical Industries, Ltd., the Chemical Engineering Division, A.E.R.E., and by the Research and Development Branch of the Industrial Group. The author has drawn freely on the relevant work of these groups.

Laboratory-scale Equipment

In developing a solvent extraction process the important parameter is the number of theoretical stages required to effect a desired transfer of components from one phase to another and in practice we find that this problem resolves itself into the determination of the number of extraction and stripping stages in a counter-current extractor. In the extraction stages the solute (*e.g.* uranium) is transferred from the aqueous phase to the solvent phase and in the stripping stages the unwanted components which have followed the uranium into the solvent are removed by an aqueous strip which joins the extraction section at the feed plate.

In most commercial solvent extraction processes the Nernst partition law

$$\frac{C''_d}{C'_c} = H.$$

is not followed because the solutions depart from ideality through association of the solute mole-

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cules and the effects of changes in composition relative to other solutes. Therefore, before a Varteressian-Fenske diagram can be drawn, the equilibrium curves of selected key components are determined by batch equilibrium of phases at various levels of relative concentration. The next stage in the design of the process is to decide, upon economic grounds, the amounts of product materials that can be allowed to flow to waste. This fixes the conditions at one end of the operating line. The next decision to be taken is the ratio of the solvent to aqueous phases in the extraction section as this determines the slope of the operating line. To reduce the quantity of solvent and to keep the dimensions of the extractors and their associated plant to a minimum, the solvent/aqueous ratio is kept as low as is compatible with a reasonable number of stages. It is also important that the operating line should not be too close to the equilibrium line because small variations in solvent-to-water ratio would then have an important effect on process efficiency.

The slope of the operating line having been chosen for both the extraction and stripping sections, the Varteressian-Fenske diagram can now be completed and the number of stripping and extraction stages determined.

This outline is a simplified version of what can be a complicated process of calculation when the conditions in the stripping section are chosen to give a build-up of one component at the feed plate and when there are several key components mutually interacting.

The next stage in the laboratory development is to determine the density, viscosity, and settling rates of the test solutions and to choose a miniature contactor which will permit the process to be tested at reasonable levels of activity on a small scale. Many devices have been developed for this purpose and as they have a general application to solvent extraction problems each will be briefly described.

Micro-airlift

This equipment, shown in Fig. 1, has been extensively used in this country and was developed for the Department of Atomic Energy by Imperial Chemical Industries Ltd. It consists of a series of glass units each comprising a mixing chamber and a settler. The solvent and aqueous phases, which flow counter-currently, enter a mixing chamber by means of the solvent and aqueous entry ports. In the chamber they are mixed and lifted by means of a deliberately inefficient Pöhle airlift into the settling chamber. Here the phases separate and continue their counter-current flow along the bank of contactors. The solvent overflow is governed by the height of the adjustable weir which is set to meet the appropriate density conditions.

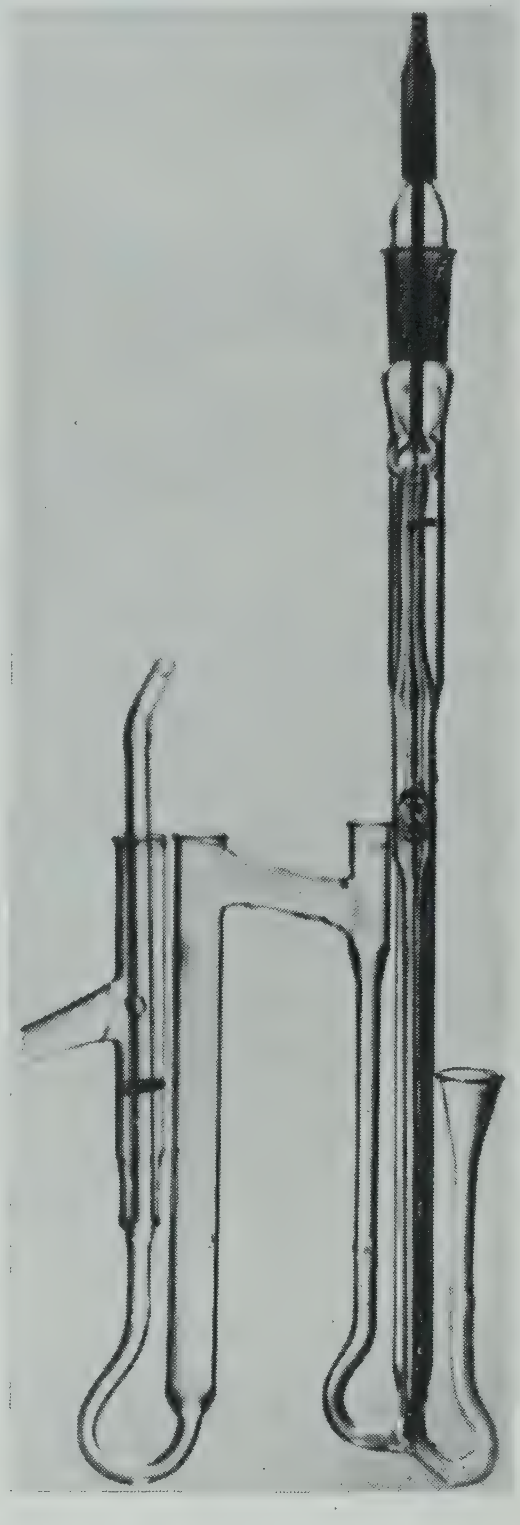

Fig. 1.—*Micro-airlift*

The Mini-mixer

This equipment, which has a smaller hold-up per stage than the air-lift unit, consists of a bank of mixers and settlers in which the mixing is carried out by stirrers. In the original design due to the Knolls Atomic Power Laboratories, U.S.A., these stirrers also lifted the aqueous phase and dispersed it into the solvent phase. In later designs used by the Research and Development Branch, Windscale, this feature has been eliminated. The fundamental theory of this equipment will be referred to in more detail when we consider the larger-scale model.

Rotary column

This device which can, in favourable systems, accommodate a large number of stages in a short height, is a rotating shaft enclosed in a column. The two liquid phases are subjected to shear forces in the narrow annulus and set up a complicated pattern of contra-rotating vortices. Although the equipment has been used for some development work at A.E.R.E. it is not favoured by the author because inter-stage samples cannot be taken to check the Varteressian-Fenske diagrams.

Flow-sheet development

By the use of such equipment the process flowsheet can be determined using less than 1 l. of feed solution. It is particularly important that the behaviour of the fission product elements should be carefully studied and it is essential that the experimental work shall be done in such a manner that true equilibrium for these materials is reached. The concentration of fission products is small, and it is quite possible to reach equilibrium in uranium and plutonium but not in fission product elements during a small-scale run of short duration. The achievement of equilibrium is best checked by frequent interstage sampling as the run proceeds ; this is easily done in the air-lift or mixer-settler type of contactor where each physical stage is distinct and approximates to a theoretical stage. In some flowsheets we have considered, 10–20 days running would be required for equilibrium in fission product built-up to be reached.

The Scale-up of Contactors

Having determined the number of theoretical stages required for each contactor, it is possible to consider the design problem for the two types of contactor discussed in this paper.

The design of packed columns

This problem, analogous to that of designing gas absorption columns, has attracted considerable interest and it is not possible in this brief survey to do justice to all the workers in this field. Pratt and his co-workers at A.E.R.E. have studied the problem of mass transfer in packed columns in considerable detail and in a series of papers[1] Pratt gives an extensive bibliography of previous work.

A packed column has two important dimensions ; the height required to accommodate the number of theoretical stages demanded by the process and the cross-sectional area required for the design throughput. These quantities are to some extent interdependent and both depend on the nature of the packing used in the column.

To appreciate the factors important in the design of a packed column it is worthwhile reviewing the fundamental theory of mass transfer. This is usually based on the Whitman two-film concept in which transfer is said to take place across two continuous films forming the interface and the driving force is the difference in concentration (or activity) of the solute molecules in the bulk of the two phases.

Expressed algebraically :—

$$\begin{aligned} dN_A &= K_c dA(C_c - C_{ci}) \\ &= K_d\, dA(C_{di} - C_d) \qquad . \qquad (1) \end{aligned}$$

If we now assume that the distribution coefficient H is constant over the section of the column we are considering, then :

$$H = \frac{C'_d}{C_c} = \frac{C_{di}}{C_{ci}} \qquad . \qquad . \qquad (2)$$

Combining equations (1) and (2)

$$\frac{dN_A}{dA} = \left[\frac{1}{K_d} + \frac{H}{K_c}\right] = C'd - Cd \qquad . \qquad (3)$$

Now by defining an overall mass transfer coefficient K_{od} (relative to the disperse phase) by the following relation :

$$dN_A = K_{od}\, dA(C'_d - C_d)$$

and substituting this relation in equation (3) we have :

$$\frac{1}{K_{od}} = \frac{1}{K_d} + H/K_c \qquad . \qquad . \qquad (4)$$

Similarly an overall coefficient based on the continuous phase can be defined by

$$\frac{1}{K_{oc}} = \frac{1}{K_c} + \frac{1}{HK_d} \qquad . \qquad . \qquad (5)$$

We may now proceed to derive the general equation for the effective height of a packed column by taking material balances over a small height dh:

$$L_c\,dC_c = L_d\,dC_d$$

But

$$L_c\,dC_c = K_c\,dA(C_c - C_{ci})$$
$$= K_c\,adh(C_c - C_{ci})$$

Integrating between $h = 0$ and $h = h$ and rearranging :—

$$\int_{C^2_c}^{C^1_c} \frac{dC_c}{C_c - C_{ci}} = \frac{K_c\,a\,h}{L_c} \quad . \quad (6)$$

The integrand on the left-hand side of the equation is commonly referred to as the number of transfer stages and the height of a transfer unit (H.T.U.) is defined as :—

$$\text{H.T.U.} = h \;.\; \left[\int_{C_2}^{C_c} \frac{dC_2}{C_c - C_{ci}}\right]^{-1} = \frac{L_c}{K_c a} \quad . \quad (6a)$$

The H.T.U. is a direct measure of the transfer rate of the solute under the local conditions in the column. When the distribution coefficient is constant and the operating line and equilibrium lines are approximately parallel, it can be shown that the H.T.U. is identical with the height of an equivalent theoretical plate (H.E.T.P.) and thus under these conditions we can express the height of column required to effect a given separation in terms of the continuous phase flow rate L_c and the mass transfer function $K_c a$.

It is in attempting to derive the function $K_c a$ from fundamental properties that we encounter the greatest difficulty, for we must be able to predict the mean droplet surface area, a, and the transfer coefficients K_c or K_d over the whole length of the column.

Prediction of interface area

It is reasonable to suppose that for a given geometry of packing, the mean droplet diameter can be related by dimensionless groups to the physical properties of the two phases and their flow-rates. If we can also produce a correlation for the volumetric hold-up of droplets we may then deduce the mean interfacial area.

Pratt and his co-workers at Harwell have carried out extensive work in this field[1] and we may summarise his approach in the following manner. Pratt defines a characteristic velocity for the disperse phase $\overline{V}_0$ as the limiting velocity when $V_c = 0$ and $V_d = \rightarrow 0$ and by considering the relative velocities of the two phases, using a modified Steinour equation for the hindered settling of droplets, he deduces the following relationship:

$$V_d + \frac{x}{1-x}\,V_c = \epsilon\overline{V}_0\,x(1-x) \quad . \quad (7)$$

To obtain the upper transition point of the packed column, *i.e.* the throughput above which the above relation no longer holds, this equation is differentiated and equated to zero, giving :—

$$V_c = \Sigma\,\overline{V}_0(1 - 2x_f)\,(1 - x_f)^2 \quad . \quad (8)$$

$$\text{for } \frac{dV_d}{dx} = 0$$

and

$$V_d = 2\epsilon\overline{V}_0\,X_f^{\,2}\,(1 - x_f) \text{ when } \frac{dV_c}{dX}=0 \quad . \quad (9)$$

Eliminating $\overline{V}_0$ from equations (8) and (9) and solving for the limiting throughput x_f

$$x_f = \frac{(L^2_R + 8\,L_R)^{\frac{1}{2}} - 3\,L_R}{4(1 - L_R)} \quad . \quad (10)$$

This relation indicates that maximum hold-up is independent of droplet size and physical properties and it can be of value in predicting the variation of the upper transition point with flow ratios and phase velocities. Once the flooding point has been determined experimentally and $\overline{V}_0$ established for a given system then the variation hold-up can be predicted for differing flow-rates.

Pratt has developed a correlation between $\overline{V}_0$ and fundamental properties and shows that the mean "volume-surface" diameter of a droplet d_{vs} is given by :—

$$d_{vs} = d_{vs}{}^0\,\frac{\overline{V}_0\epsilon x}{V_d}$$
$$= 0{\cdot}92\left(\frac{\gamma}{\Delta\rho g}\right)^{0.5}\left(\frac{\overline{V}_0\epsilon X}{V_d}\right) \quad . \quad (11)$$

where $d_{vs}{}^0$ is the "characteristic droplet diameter" defined as the mean volume-to-surface ratio when $V_c = 0$ and $V_d \rightarrow 0$. Now as the ratio of surface to volume of a sphere is 6 the area of contact between the two phases in a column is given by :—

$$a = 6x/d_{vs} = 6V_d/d^0{}_{vs}\overline{V}_0 \quad . \quad (12)$$

Thus by determining $\overline{V}_0$ for a particular packing it would appear that the interfacial area of the droplets for any flow-ratio can be predicted from a knowledge of phase densities and interfacial tension.

However, there are serious difficulties in that Pratt has shown that the interfacial area calculated on this basis does not always apply in the presence of an organic solute or when the phases are not mutually saturated. He finds that the droplet size and $\overline{V}_0$ increase in the absence of mutual saturation and that the H.T.U. found in practice is considerably greater than would be predicted by this relation.

The final factor to be evaluated before the column heights can be predicted is the overall mass transfer coefficient K_{od}, and in this brief survey it must suffice to say that although experimental values of K_{od} can be determined by transfer-cell techniques it is difficult to predict a value of K_{od} which can be applied to column design because of such variables as internal circulation in the droplets, droplet oscillation induced by mass transfer, and interfacial resistence due to adsorption of impurities. The approach forced upon the chemical engineer is the semi-empirical one of determining the H.E.T.S. or H.T.U. on a small diameter column and then of scaling-up and this will now be considered.

Scaling-up column diameter

Ballard and Piret[2] describe three régimes of flow in a packed column; flooding, phase reversal, and "slugging" and their measured transition points correspond closely to the flooding points given by equations (7), (9), and (10). Pratt recommends that the column be designed for operation at flow rates somewhat below the flow transition points and at a hold-up of some 15–25%. Using these criteria we can calculate from equation 7 the desired hold-up if x_f has been determined by a model test.

From this we can obtain $\overline{V}_0$ for which Pratt proposes the following semi-empirical correlation:—

$$y = 1 - \frac{y}{z}\left[1 - \frac{e}{y}\right] \quad . \quad . \quad (13)$$

where

$$y = \frac{\overline{V}_0}{V_t}\,[1 - e^{-7.2}\,D_c]$$

and

$$Z = \frac{\Delta\rho g\bar{s}}{\rho d\,V_t^{2}}.$$

The mean path length $= \bar{s}$ is defined by:—

$$\bar{s} = 0{\cdot}38\,d_p - 0{\cdot}92\left[\frac{\gamma}{\Delta\rho g}\right]^{\frac{1}{2}}$$

Scaling-up column height

Combining the usual expression for H.T.U. (equation (6*a*) with equation (12)) we have

$$\text{H.T.U.}_{\cdot(d)} = \frac{d_{vs}{}^{0}\,\overline{V}_0}{\sigma\,K_d} \quad . \quad . \quad (14)$$

$$\text{H.T.U.}_{\cdot(c)} = \frac{a^{0}\,ys\,\overline{V}_0 V_c}{\sigma K_c\,V_d} \quad . \quad (15)$$

Thus K_d and K_c are dependent on the droplet velocity relative to the continuous phase and H.T.U.$_{\cdot(d)}$ should be constant whilst H.T.U.$_{\cdot(c)}$ is proportional to V_c/V_d. However, it is found that the "self shielding" of droplets increases with hold-up and introduces a factor X^m into the following empirical correlation which Pratt finds fits his data to ± 15%.

$$\frac{Kd_{\alpha}\,\epsilon\,X^{1+m}}{V_d} = 6{\cdot}4 \times 10^{-5}\left[\frac{d^{0}{}_{vs}\,\overline{V}_0\,\rho d}{\mu d}\right]^{0.5} \left[1 + \frac{V_c X}{V_d(1-x)}\right]^{1.5} \quad . \quad (16)$$

where

$$m = 0{\cdot}45 - 0{\cdot}2\,d_p.$$

A similar correlation holds for K_c.

To account for longitudinal mixing, a form of internal recirculation of phases leading to an increase in H.T.U., Pratt uses a factor F_m which is experimentally determined for a particular system.

Pratt found that experimental values of H.T.U.$_{oc}$ for butyl acetate-water-acetone system were 30–60% lower than those predicted from equations (14), (15), and (5) and suggests that micro-oscillation of the droplets is responsible for the difference. However, the method gave considerably better agreement for the transfer of uranyl nitrate between water and M.I.K. if the effect of interfacial resistance were taken into account by using values of interfacial resistance determined in transfer cell experiments.

The design of the Windscale columns

The foregoing survey of the work by Pratt and his colleagues shows the difficulties in designing a packed column from fundamental data. With the differential transfer of several mutually interacting solutes, *e.g.* uranium, nitric acid, and plutonium, the problem becomes too complex for a practical solution to be achieved on these lines. The course that must be chosen is that of determining the value of the H.T.U. for a key component such as uranium on a model system and then scaling-up to the final design.

When the design of the Windscale columns was undertaken, the work at A.E.R.E. had not advanced very far and could not be used as a basis for scaling-up. Furthermore, as the height of the columns fixed the height of the massive concrete radiation shield, it was imperative that the design estimates should be reliable : modification while building was in progress could not be accepted.

The approach we made was therefore more direct but it has thrown some light on the problems involved in packed column design.

The Windscale columns were sized in the following manner :

(1) Determination of distribution data for uranium, plutonium, and gross fission products by batch equilibration.

(2) Calculation of the number of theoretical stages required to effect efficient separation of uranium and plutonium.

(3) Determination of the H.E.T.S. and flooding points for uranium in 2 in. packed columns ; the extraction and stripping sections being treated separately.

(4) Calculation of the effect of minor flowsheet changes (solvent/aqueous ratios) on flooding rates from the relation $V_c^{\frac{1}{2}} + V_d^{\frac{1}{2}} = \text{const.}$

(5) From the flooding data, prediction of column diameter for full-scale operation on a direct area scale-up.

(6) Determination of H.E.T.S. for uranium on the extraction and strip sections of full-size columns.

It will be appreciated that this approach gave firm figures for the uranium extraction and, by comparison, reasonably firm figures for plutonium (the equilibrium data for which were available). The determination of the equilibrium data for the mixed fission products under all the conditions of the column was not possible at that time but it was found that the fission products distribution coefficients could be roughly grouped at three levels : 0·3, 0·1, and ·005.

The stripping section of the first column was designed to give a decontamination factor* of 10^4 for all fission products for which the distribution coefficients were 0·1 or less. This involved the assumption that the H.E.T.S. for the fission products was of the same order as that of uranium. The actual values of the decontamination factors obtained could not be known until the plant was fully active : the design predictions were largely confirmed although some fission products, *e.g.* ruthenium and zirconium were not removed to the extent originally desired. Indications that this would be so were obtained in laboratory work and provision had been made for dealing with these isotopes in subsequent stages of the process.

The inactive pilot-plant work was confined to studies on systems having flow ratios approximately those required by the flow sheet and the effect of minor variation in flow ratio on the flooding point was predicted from the simple relation given by Breckenfeld and Wilke[3] :—

$$V_c^{\frac{1}{2}} + V_d^{\frac{1}{2}} = \text{const.}$$

where the constant is determined for a given column and packing.

The value of H.E.T.S. at flow-rates equivalent to 60–70% of the flood point rates was next determined for the 2 in. column for the extraction and stripping sections separately and then, finally, combined at the extrapolated height. This prodecure was permissible because the conditions in the strip section were chosen to give the same distribution coefficient for uranium as in the extraction section and build-up at the feed plate did not occur.

After the flooding point of the 2 in. columns had been determined, the required diameter of the full-scale column (about 12 in.) was predicted on a direct area scale-up and a full-scale extraction section erected. The flooding point was then determined and the H.E.T.S. measured arbitrarily chosen to be 60–70% of flood point rate.

It was found that the H.E.T.S. was greater by a factor of 1·5 to 2 than that predicted from the 2 in. column. On the assumption that this increase was due to maldistribution or " channelling " of the phases within the column, perforated distribution plates, fitted with 1 in. diameter downcomers, were placed at intervals along the column. An improvement in performance was obtained but even so the values of H.E.T.S. found on the 2 in. column were not achieved. Attempts were made to improve the H.E.T.S. by fitting spray distributors at the feed point and solvent inlets. Changes in packing size were made to determine if an improvement in H.E.T.S. could be achieved. These showed that an optimum size existed (for Lessing rings) each column. It is interesting to note that in the smaller sizes the droplets of disperse phase clung to the packing and did not break free.

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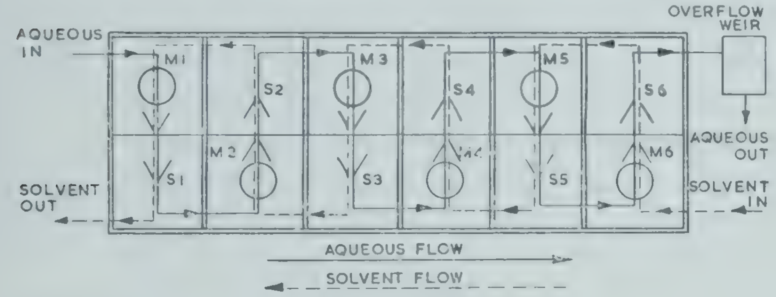
* Defined as the ratio of activity in the feed to activity in the solvent extract.

In this manner, the final design dimensions of the columns and their packings were determined and subsequent experience on the production plant has confirmed the reliability of the method.

The importance of suspended solids in reducing packed column performance has long been known; the effects are due to a reduction of voidage volume leading to premature flooding. Transfer rates may also be affected by the formation of "stable" emulsions leading to internal re-circulation. Allowance should always be made for effects of solid impurities by working well below the flood point and providing one or two additional theoretical stages.

Scaling-up mixer-settlers

This type of extractor has the advantage that the volume of shielding required for radio-active plant is less than that of a packed column and in plant design considerable economies can be effected. Fundamentally a counter-current extractor of the mixer-settler type is represented by Fig. 2, in which the solvents flow counter-currentwise through the bank and mixing is co-current.



M = Mixer
S = Settler

Fig. 2.—*Flow pattern in counter-current mixer-settler*

There have been many types of mixer-settler described in the literature and they may be classified into those requiring inter-stage lifts (*e.g.* pumps) and those in which the counter-current flow is maintained entirely by the hydrostatic head generated by the density difference of the phases. In the former group we have the Mensing, Edeleanu, and K.A.P.L. type of contactor, whilst the latter group is exemplified by the Holley-Mott and Windscale types.[4, 5, 6, 7, 8]

It is only proposed to discuss here the scaling-up of contactors of the Windscale type as we have little experience of the pump-lift type; in the pump-lift contactors the hydrodynamic factors affecting pumping rates can have a major effect on the stability of operation and it is usual to incorporate instrumentation to maintain interface levels.

For systems in which no significant change in either the volume ratio or density ratio of the two phases occurs in the process, it has been shown by Williams and Lowes[8] that the height of the interface can be predicted by taking a simple hydrostatic balance over the system. Fig. 3 shows a mixer with its upstream and downstream settlers. Taking hydrostatic balances about the mixed phase port M and the aqueous port A it can be shown that

$$h_2\rho_m + B\rho_m = h_1\rho_s + B\rho_s - x\rho_s + x\rho_a \,. \qquad (17)$$

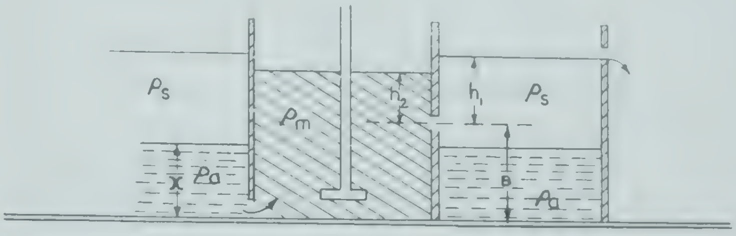


Fig. 3.—*Hydrostatic balance in mixer-settler*

But the density of the mixed phases, ρ_m can be expressed as :—

$$\rho_m = f\rho_a + (1 - f)\,\rho_s \,. \qquad (18)$$

where f is the volumetric fraction of the aqueous phase in the mixer. From equations (17) and (18) we find that

$$x = Bf.$$

Using this simple equation the height of the interface can be predicted and the design height of the mixed phase port selected.

The scaling-up of the box mixer-settler then resolves itself into the scaling-up of the mixer, the settler, and the mixed-phase and aqueous ports.

Scaling-up the mixing chamber

The important parameter in scaling-up the mixing chamber is the mean residence time of any particular volume of reactants and this time is determined on small-scale laboratory trials in which the transfer rates are measured against time. For the systems to which our contactors have been applied a residence time of 1–2 min. is sufficient for stage efficiencies of 100% if good mixing can be assured.

A flat two-bladed paddle has been chosen which eliminates lift and gives the minimum transfer of kinetic energy through the aqueous and mixed phase ports. To assist in this, each port is fitted with a vertical baffle. Mixing is increased by the use of four narrow vertical baffles off-set from each wall of the mixing chamber.

We have found that by scaling-up the mixing chamber to give the same residence time in a geometrically similar plant to that of the small-scale model the predicted results have been obtained on equipment having throughputs 100–200 times that of the model. The prediction of power input into the mixer can be made in several ways. For unbaffled tanks White and Summerford[9] use the empirical relation :—

$$P = cL^3 \rho N^3 D^{1.1} \,.\, W^{0.3} \,.\, H^{0.6}$$

where $P =$ power input, $L =$ paddle length (ft.), $N =$ paddle speed (r.p.m.), $\rho =$ density (lb./cu. ft.), $W =$ paddle width, $H =$ liquid depth and c is a power coefficient for which they give a correlation against a modified Reynolds number $\Bigl(\frac{L^2 \,.\, N \,.\, s.}{Z}$, where $Z =$ absolute viscosity lb./ft. (sec.).$\Bigr)$ For baffled tanks the scaling-up of power consumption is more difficult and the power may be several times that predicted by White and Summerford. Design data for specific designs are given by Hixson and Bawn[10], and Mack and Kroll[11] and can be used as a guide.

Olney and Carson,[12] give a generalised correlation :—

$$P = \alpha L^{4.7} N^{2.85} \rho^{0.85} \mu^{0.15}$$

where α is dependent on impeller design and the authors give a nomogram enabling the power requirements of several types of stirrers, including flat paddles, to be estimated. Miller and Mann[13] conclude that for mixing two immiscible liquids with paddle stirrers the criterion for scaling-up of geometrically identical systems should be that of equal power input per unit volume of liquid. With due allowance for the differing transmission losses, this simple method of scaling-up has been found to be satisfactory.

The power requirements for the large contactors now in use at Springfields factory are small and a conservative approach was used in estimating the power required.

The scaling-up of settlers has been done entirely on residence time and geometrical similarity and this method has been satisfactory in practice. The fundamental theory of settling in a continuous flowing system of this type is, as far as the author is aware, a new field and it is hoped to undertake some systematic studies in this field at Windscale, using the approach made by Steinour to the settling of particles and imposing the horizontal phase velocities upon the settling velocities of the droplets. In this way the profile of the settling emulsion may be determined. However, we can expect interference due to flow reversal at the downstream wall and emulsion stabilisation by solid impurities.

An over-riding factor in any attempt to scale-up settler sizes from laboratory models is the presence of impurities which collect at the settler interfaces and may, in certain systems, form stable emulsions. As these can vary with the quality of the feed material, it is advisable to introduce a safety factor in settler sizes, and volumes of 50–100% excess have been employed in certain units where considerable variation of fuel types can be expected as reactor development proceeds.

The design of the aqueous and mixed phase ports has been done on the basis of the usual formula for flow through submerged orifices; this has been shown to be satisfactory for the aqueous phase ports but some interesting viscosity effects have been noticed by Williams and Lowes when studying the mixed phase port. These are associated with phase reversal in the emulsion and have led to deliberate over-design of the mixed phase port. Since this is protected by a vertical plate baffle to reduce the pumping effect of the impeller, no ill effects have been observed on the operation of the contactors.

Using the approach outlined above it is possible to scale-up directly, without pilot plant studies, from contactors having a throughput of a few litres per hour to ones capable of handling a cubic metre per hour or more.

Even so it will be appreciated that there is still room for further work in scaling-up the hydrodynamics of mixer-settlers. We have carried out very little work on the dynamics of mass transfer in the mixers and no doubt results of some value would accrue if the methods proposed by Vermulen and MacMullin and Weber were used. None-the-less, for systems in which the transfer takes place rapidly, there is little economic value in such research because the differences in volume of mixer which would be predicted by the direct approach of scale models and by allowance for non-homogeneous residence times will be small.

Conclusion

From our experience in the field of scaling-up solvent extraction process equipment it can be said that the scaling-up of the packed columns with any degree of accuracy is difficult and requires considerable effort, although recent work has led to a greater understanding of the problem. The scaling-up of mixer-settlers of the Windscale design is found to be comparatively simple and satisfactory techniques are available which eliminate the need for pilot plant trials.

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Symbols Used

αA = element of interfacial area.
a = interfacial area per unit volume of column.
C'_d = equilibrium concentration of solute in disperse phase (lb. mol./cu. ft.).
C_d = concentration of solute in disperse phase (lb. mol./cu. ft.).
C_{id} = concentration of solute in disperse phase at liquid interface (lb. mol./cu. ft.).
C_c = concentration of solute in continuous phase (lb. mol./cu. ft.).
C_{ci} = concentration of solute at interface of the continuous phase (lb. mol./cu. ft.).
d_{vs} = mean volume-to-surface diameter (ft.).
H = partition coefficient.
K_c = mass transfer coefficient (from continuous phase) (lb. mol./hr. sq. ft.).
K_d = mass transfer coefficient (from disperse phase) (lb. mol/hr. sq. ft.).
K_{oc} = mass transfer coefficient (overall) (lb. mol./hr. sq. ft.).
K_{od} = mass transfer coefficient (overall) (lb. mol/hr. sq. ft.).
L_c = flow rate of continuous phase (lb. mol./hr.).
L_d = flow rate of disperse phase (lb. mol./hr.).
L_R = flow ratio (L_d/L_c).
N_a = number of lb. mol. of solute transferred in unit time.
$\overline{V}_0$ = characteristic droplet velocity, *i.e.* mean velocity when $V_c = 0$ and $V_d \to 0$ (ft./hr.).
V_c = superficial velocity of continuous phase (ft./hr.).
V_d = superficial velocity of disperse phase (ft./hr.).
X = fractional volumetric hold-up in packing voids.
X_f = fractional volumetric hold-up in packing voids at flood-point.
Σ = fractional voidage of packing.
ρ = density (lb./cu. ft.).
γ = surface tension (lb./sq. hr.).

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DISCUSSION OF PAPERS PRESENTED AT THE SECOND SESSION

Dr. H. R. C. PRATT said in a contribution read by Dr. J. D. Thornton :

Dr. Reman has given an interesting account of the methods presumably used for the design of rotary disc contactor by his company ; incidentally, the original development of this contactor was due to Dr. Reman himself. His present paper appears to be an extension of an earlier one[1] and it was gratifying to see new data for a large diameter unit.

It appeared surprising that Dr. Reman had expressed his correlation of flood-point and stage efficiency in terms of dimensional and not dimensionless groups, and Dr. Pratt could not accept the explanation given early in the paper. It is true that some terms, such as the acceleration due to gravity, were constant and could be neglected in some circumstances ; nevertheless it was useful to have the complete dimensionless groups available, thus making it easier to use any desired set of units.

The flood-point correlations in Figs. 2(A) and (B) were peculiar in that some of the data referred to non-flooding conditions. Presumably these referred to actual units designed to operate at about 50% of the flood-point, in which case presumably the curve shown was intended to represent a safe upper operating limit. Dr. Reman was presumably familiar with the alternative method of correlation, employing the " characteristic velocity " of the droplets, which had been developed at Harwell and shown to be applicable to a wide range of contactors, including the rotary disc type. They had found that the data obtained for a contactor of this type were well correlated to within about ± 20%.

The correlation of stage efficiency was much more difficult than that of throughput, but Dr. Pratt was not at all happy about the approach used in the paper. It was true that the droplet size would be determined in some manner by the energy input, but this was surely not true of the mass transfer coefficient. For large droplets the latter was determined, at least partially, by considerations of eddy transfer due to the internal circulation known to take place, but at high stirring speeds, where the droplets became very small, it was almost certain that in practice they would become effectively rigid, so that molecular diffusion would then be the controlling factor. It would be interesting to know why Dr. Reman had not incorporated the extraction factor terms in the correlation shown in Fig. 9, since this should bring all the data on to a single curve.

Dr. Pratt wished to know the maximum diameter of a column of this type which Dr. Reman would consider it safe to build at the present time, and also whether he would consider this type of contactor suitable for use when a moderate number of theoretical stages, 10 to 15, was required.

Referring to Mr. Warner's paper, Dr. Pratt agreed that at the time when the Windscale solvent extraction plant was being designed, the Harwell design methods for packed and other types of extraction column had not been available, and in consequence a lengthy and empirical procedure had necessarily to be adopted ; but, although mixer-settlers were attractive for radioactive work because the small height reduced the shielding costs, he could not agree that the scaling-up of mixer-settlers was as simple as Mr. Warner claimed.

It was true that the mixer-settler comprised a number of discrete units and that in some circumstances each unit had an efficiency of approximately one theoretical stage, but the same problems were encountered in the design of mixer-settlers as in the design of columns, so that for an accurate design it was necessary to determine, or if possible predict, the capacity and the stage efficiency. The capacity was determined by two hydrodynamic factors in the settling chamber : the rate of settling of the droplets, and the rest-time necessary for coalescence. Both these factors depend upon the droplet size and therefore on the power input. Knowing the droplet size, the first was determined by the usual friction-factor curve for spheres, while the latter has been explained by Gillespie and Rideal[2] as being due to the rate of drainage of the film of liquid trapped between the drop and the interface. The efficiency, on the other hand, was a function of the rate of molecular or eddy diffusion or both, and was affected by droplet size, droplet coalescence, and so on exactly as in the case of column extraction. Some success had been obtained by Vermeulen and his co-workers at the California Institute of Technology on the determination and correlation of interfacial area in liquid-liquid mixers, but much further work would be necessary before the performance of mixer-settlers could be predicted with confidence.

It was perhaps true that the mixer-settler appeared to be a more flexible instrument than column contactors, but only if a large excess capacity was built in. In the case considered it was possible, once the plant was built, to operate at a higher stirring rate than that expected, if it was found that the stage efficiency was low. Such a procedure, however, was also applicable to mechanical types of column contactor, but should be avoided wherever possible, because it would lead to unnecessary capital expenditure.

Dr. J. D. THORNTON said that there was no reference in the text of Dr. Reman's paper to the method by which the curve in Fig. 3 had been obtained, and it would be interesting to know how in fact it had been calculated. With regard to the correlation of the mass transfer data in Fig. 9, he found it difficult to understand why a correlation of column efficiency had been sought in terms of the energy input only. The observed overall mass transfer coefficient was a composite function and only the area term, a, would be expected to correlate with the energy input. The coefficient K, on the other hand, could be correlated[2] by a modified Stanton number-Reynolds number relationship of the form

$$\left(\frac{K}{v}\right) \propto \left(\frac{d \,.\, v \,.\, l_c}{\mu_c}\right)^m$$

when d and v are the mean outlet diameter and relative velocity respectively. It would appear that the correlation was incomplete as presented, and he would suggest that in general a closer correlation could be obtained by allowing for variations in K by means of the Stanton number-Reynolds number relationship.

The curves of efficiency against rotor speed in Fig. 5 exhibited maxima at various rotor speeds, and it had been suggested that this was due to the mixing effect in the column. There was, however, an additional phenomenon which gave rise to this effect, namely the onset of a molecular-diffusion-controlled transfer process within the droplet as the droplet size decreased. It was important to distinguish between these two effects, and he would like to know from Dr. Reman the order of the droplet size at which these maxima occurred.

Dr. Thornton said that after reading Mr. Warner's paper he felt that the author had been a little pessimistic in saying that the scaling-up of packed columns with any degree of accuracy was difficult. This was not strictly true. The difficulty in the design of column-type contactors lay in the prediction of flood-point and H.T.U. characteristics when complicating factors such as interfacial effects which gave rise to droplet coalescence, were present. Once the magnitude of these effects had been determined by carrying out a limited number of H.T.U. and hold-up determinations in a laboratory-scale contactor, there was no difficulty in predicting the stage height and flood-point of the large scale unit. Any difficulties lay, therefore, in the initial prediction of characteristics peculiar to the extraction system, and not in the scaling-up of column-type contactors once such factors had been determined experimentally.

Mr. P. Eijsberg, commenting on Dr. Reman's statement that by increasing the power input per unit mass the efficiency of the contactor increased, said that for all types of extraction apparatus the trend was to increase the efficiency as much as possible without emulsification. Most of the author's data involved energy inputs of a high magnitude ($N^3 R^5/HD^2 = l - 10$). The advice was given that for the scaling-up of efficiency it was desirable to keep the energy input low, in order to use the proportionality of the efficiency and the diameter. There seemed to be a contradiction here. Would not it be cheaper to work with higher energy inputs—the total energy seemed rather low—even if the above-mentioned proportionality could not be used? Or were there other reasons for this choice?

Dr. Reman considered the contactor as equivalent to a number of ideal mixers in series, with the phases in counter-current flow. Was anything known about the residence time-distribution curve for both phases, especially compared with those of a number of ideal mixers? Especially for small phase ratios the maximum capacity increase was considerable. Could Dr. Reman say anything about the behaviour of the contactor with regard to capacity and efficiency when liquids with a great difference in specific gravity were used, and at low phase ratios?

Mr. Th. W. Willemse said that the empirical and semi-empirical relations mentioned in the paper by Dr. van de Vusse arose from experiments described in the literature, and one felt happy that such a large part of the field was covered by them with, apparently, some reliability.

Some doubt arose, however, where only partial experimental evidence supported the theory given under the heading "Isotropic turbulence". In particular, Mr. Willemse's attention had been attracted by the statement on p. S43 that particle velocity was equal to settling velocity minus fluid velocity. This was no doubt correct, but one might not feel entirely on safe ground when applying the diffusion concept in cases where large fluid velocities occurred, and thus a substantial fraction of the lifting force was derived from the large-scale fluid motion.

It might be that in practice one should ask not where the diffusion concept failed but where the border lay between lifting by turbulence and lifting by pumping action, at least if it were correct to translate the expression "non-isotropic turbulence" as "pumping action". As an example, he would mention Dr. van de Vusse's statement on p. S45 that "paddle stirrers need a 4- to 5-fold higher power consumption than propeller stirrers", if used as a means for suspending particles. Was the author quite sure that the power needed to maintain the particles in suspension—or, in other words, to pump them up in a fluid vortex—was a measure for scaling-up in the case of a paddle stirrer? Would not a large part of the lifting action in this case be derived from turbulence? An argument for this view might be that Dr. van de Vusse stated elsewhere[4] that the power consumption required to ensure a certain mixing time, when mixing miscible liquids, was equal for propeller stirrers and paddle stirrers. This would mean that a paddle stirrer was an efficient mixing implement but an inefficient pump, as might be inferred from its shape.

On the other hand, with jet-mixing of miscible liquids, a power consumption four-fold higher would be required, so that a jet was an inefficient mixer ; but he would suggest that it could be an efficient pump for solid particles if a suitable geometry was chosen, for example with the direction of the jet at an angle to the bottom of the tank. The turbo-mixer might occupy an intermediate place in both respects ; but could the author say whether the various kinds of vibrational mixers would, depending on the shape of the stirring element, behave in a similar way to that of the mixers mentioned already, or did he suspect that unforeseen influences of the vibration itself, as opposed to rotation, might occur, for instance at high frequency?

Dr. R. LESSING said that he had been gratified to hear Dr. Thornton say that scaling-up of packed columns could be done with greater ease and accuracy than Mr. Warner had assumed. The widespread use of packed columns proved that they gave a certain degree of satisfaction to industry in general, despite the scaling-up difficulties. An outstanding example was the work of the United Kingdom Atomic Energy Authority which demonstrated that very large plants could be successfully designed on the basis of research apparatus made on the smallest scale.

When scaling-up empirically it was essential that every factor which from experience was known to play a part should be considered. Some of the dimensional proportions which would apply to the large plant could not be reduced to the scale which would fit the experimental one.

By way of example, Dr. Lessing referred to a passage at the foot of p. S58 of Mr. Warner's paper. Dealing with small columns of, say, 2 in. diam. an optimum size of packing rings had been observed. The relation of size of packing unit to diameter of tower was, of course, the most important factor in design, in view of the effect on the distribution of both liquid and vapour in the case of distillation or scrubbing, or of the continuous and dispersed phase in the case of solvent extraction.

Another factor was the wall effect. When regular packing elements in irregular arrangement touched the wall of the vessel, deflection, and hence redistribution, became inoperative, and liquid might run down directly along the wall surface.

Mr. Warner had stated that using 1 in. pipes as downcomers in a 2 in. column gave an improvement in performance, but that even so the theoretical possibilities had not been achieved. Dr. Lessing wondered whether the observed deficiency might have been due to an enhanced wall-effect on the outside of the downcomers superimposed on that on the inside of the column itself.

A small point might be mentioned concerning the droplets clinging to the surfaces of the packing elements. This tended to occur in narrow columns, mainly because the bubbles were too large in relation to the voids within and between the packing elements. If, as he assumed, $\frac{1}{2}$ in. rings had been used, the bubbles at the velocity chosen might have been impeded by excessive impact on the surfaces of the packing. There was, however, also the effect of viscosity, vapour tension, and, most important, the surface forces of the solid material itself. As long ago as 1921 he had pointed out[5] the importance of these factors. He had observed indications even at that time—and on many occasions later—that the physical and chemical properties of the material of which the elements were made had differing effects on the various liquids passing through the column, causing them to be either attracted to or repelled from, the surfaces. Mr. Warner's observation of the peculiar clinging of droplets to the surfaces might possibly be explained in that way.

Mr. W. S. VAN DER LAAG said that Mr. de Gelder's paper was in two parts, the first discussing the model laws and the second giving the results of the author's calculations. In the first part the author stated that there were three similarity criteria which had to be considered : a Reynolds number, a Froude number, and a coefficient which was stated to be the ratio of specific gravities of the dispersed and continuous phases. It was not clear, however, how the size of the particles to be separated was taken into account in this picture, and it was difficult to see how the Froude number could be a similarity criterion, since it contained the acceleration of gravity, which usually played no part in the separation mechanism in cyclones. The latter could be clearly seen from equation (3) in the paper, which did not contain the accleration of gravity. It would be interesting to know, therefore, why the author included the Froude number as a similarity criterion.

In discussing the calculations which the author had performed it would be interesting to know the underlying assumptions, and in particular it was important to know the range of variables within which the theoretical results had been confirmed by experiments. It was not clear how the influence of wall roughness could be seen in equations (2) and (3). It was well known that wall roughness has an effect on both pressure drop and separation efficiency. It would also be interesting, because of the importance of the results given in the paper, to have the derivations of the formulae given by the author.

Mr. W. S. van der Laag later wrote :

One of the points raised earlier during the discussion of this paper may be considered somewhat closer. It concerns the question whether in scaling-up, taking geometrical similarity and using the same fluid and particle substance, the Froude number should be kept constant if the same separation is to be obtained. Considering Mr. de Gelder's equation (3), which expresses the size of the largest particle which cannot be separated in a cyclone, it is easily seen that this may be written as follows :

$$d_0^2 = \frac{18\,\eta}{2\pi\,(\rho^1 - \rho)}\,\frac{D_2^2\,b_0}{V_0\,D_1(D_1 - \xi\,D_2)} \quad . \quad \text{(a)}$$

since the figure 0·349 in the paper apparently equals $\pi/9$. In scaling-up under the conditions mentioned above it follows from equation (a) that

$$d_0^2 = \text{constant} \;.\; \frac{\eta\,D_1}{(\rho^1 - \rho)\,V_0} \quad . \quad \text{(b)}$$

in which η is the dynamic viscosity of the fluid. Therefore, in order to obtain equal separation diameters d_0, V_0 should be taken proportional to D_1. However, scaling-up under the condition of constant Froude number would require V_0 to be taken proportional to the square root of D_1.

Exactly the same result as expressed by equation (b) follows, of course, if the particle to be separated is characterised not by its diameter, d_0, but by its settling velocity under the action of gravity, v_s. Using Stokes' law, equation (a) may be written as

$$V_s = \frac{g}{2\pi}\,\frac{D_2^2\,b_0}{V_0\,D_1\,(D_1 - \xi\,D_2)} \quad . \quad \text{(c)}$$

and it is seen, again taking geometrical similarity, that

$$V_s = \text{constant} \;.\; \frac{g\,D_1}{V_0} \quad . \quad . \quad \text{(d)}$$

once again expressing the requirement that V_0 be proportional to D_1 for equal separation.

From this it would follow that larger cyclones should be operated under higher Froude numbers than geometrically similar smaller ones for equal separation, the Froude number itself being proportional to the linear dimensions of the cyclone. It would seem that an incorrect similarity criterion (Froude number) has been given in the paper as a consequence of the inclusion of a physical quantity (g) which in the majority of cases plays no part in the separating action inside a cyclone.

It may be mentioned that the same result as above can be derived from expressions analogous to equation (3) in the papers which have been given by various authors[6, 7, 8] taking a different approach.

Mr. D. Bradley extended the discussion of Mr. de Gelder's paper to the hydraulic cyclone, because, he said, there were very many points of similarity between the gas and the hydraulic cyclone, though there were also important points of difference. The author had clearly shown that complete dynamic similarity between large cyclones and model cyclones was impossible to achieve, due to the incompatibility of the Reynolds and Froude groups. It was therefore necessary to resort to theoretical or empirical correlations, or both, in calculation, and this was equally true for the hydraulic cyclone. The author had shown, however, that the laws of similarity were still useful, in conjunction with these calculations, for scaling-up from model tests.

In these methods of calculation Mr. Bradley had been surprised to see the introduction of so many empirical constants which did not appear to have any physical significance. This could be avoided by the use of a more direct approach, for example that of Stairmand,[9] for the calculation of pressure-drop. Incidentally, decrease in pressure-drop with increase in the viscosity of the fluid in the cyclone is also predictable by this method, if the consequent change in the peripheral velocity is taken into account.

The usefulness of Mr. de Gelder's approach was not contested, and by way of example Mr. Bradley had calculated the pressure-drop for the first example given in Table IV of the paper by Stairmand.[9] The observed pressure-drop was 14 milliatmospheres ; that calculated by Stairmand's technique was 11 milliatmospheres, while that calculated by de Gelder's method was 13·7, which was very satisfactory.

On a point of detail, Mr. de Gelder referred in his paper to the use of a cylindrical surface to define the equilibrium grain. Many workers have shown that the equilibrium envelopes in a cyclone cone are themselves conical. Did this mean that separation was considered to be completed in the cylindrical portion of the cyclone, or was circulation in the cone ignored in the author's approach to the subject?

It would be interesting to have the author's views on how far the relationships which he gave could be extended to the hydraulic cyclone, even though there were important differences, one of which was compressibility. Another important difference could readily be seen by using the author's expressions. If the pressure loss was equated to the centrifugal-head loss the velocity change at the inlet to the cyclone could be readily calculated for any value of the flow factor. It would thus be seen that the gas cyclone usually operated with the spinning speed at the periphery greater than the average speed in the inlet duct. The hydraulic cyclone, on the other hand, mainly operated with an inlet velocity loss ; there was an average speed in the inlet duct greater than at the periphery. This change in velocity at inlet in cyclone operation did not appear to be widely appreciated. It was also related to the fact that the hydraulic cyclone could operate with a Reynolds number less than the specific, as defined by the author. Did this mean, in the author's opinion, that the conditions of flow in the hydraulic cyclone were governed wholly by boundary layer considerations, or were his values of the factor G inapplicable in this instance?

Dr. F. V. A. ENGEL said the Mr. de Gelder characterised the efficiency of separating performance by a critical Reynolds number and also mentioned that the physical significance of the critical number was that the whole of the fluid which left the cyclone came from the boundary layer and was, therefore, not subjected to the separating forces of a vortex. The question arose of whether this critical Reynolds number was related to some local true critical Reynolds number or if this was merely a convention. In the latter case it would be unfortunate to use a term which should only be employed in cases where, somewhere in the fluid stream, a critical Reynolds number in the true meaning of fluid dynamics occurred. If this was so, it would be preferable to replace the term " critical Reynolds number " by " specific Reynolds number ". This would prevent further confusion in terminology*.

In the case of nozzle flow,[10] for instance, reference might be made to a critical Reynolds number related to the main fluid stream of, say, 150 000, or even 500 000. The length dimension of this Reynolds number referred to the diameter of the narrowest section of the nozzle. The meaning of this statement was that the predominant influence of viscosity for larger values of Reynolds numbers had ceased to exist. The high values of Reynolds numbers referred to, however, could be correlated to a specific Reynolds number of, say, 3000 in the boundary layer. Beyond this critical value the laminar boundary layer had broken down and no longer acted as a displacement layer.

It would be very helpful if the author would elucidate his statement. It would be of particular interest to know why the Reynolds number became so predominant, whilst the general conception might be that the Froude number had a particular bearing on the transport mechanism of rigid particles entrained in an air stream. An even more important question was whether or not for various model scale ratios true dynamic similarity existed with respect to the boundary layer. For comparatively simple structures such as nozzles Dr. Engel thought that this was not the case.

Dr. G. H. REMAN expressed surprise that Mr. B. F. Warner had stated that the rotary column was not favoured because the inter-stage sampling was so difficult. In general, he said, rotary columns were very efficient, and he wondered whether this sampling difficulty could be put forward as an excuse for not using this equipment on a commercial scale.

The author also stated that the H.T.U. (height of a transfer unit) was identical with the height of an equivalent theoretical plate (H.E.T.P.). This held true only if the extraction factor was unity. The more general formula is

$$\text{H.E.T.P.} = (\ln E)/(E - 1)\ (\text{H.T.U.}),$$

E being the extraction factor.

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* The author has accepted Dr. Engel's suggestion.—*Hon. Ed.*

He had been surprised to find that the work of Rushton, which gave excellent data on the power input for stirred vessels, was not mentioned. They had always had successful experience with these data. On the design of settlers, a great deal of work had been done in the Shell Group about ten years ago by J. Heldman. From that work it appeared that one of the most important variables in the design of a settler was the ratio of the interface area of the settler divided by the load on the settler.

Mr. D. C. F. PRATT agreed that all the criteria mentioned on p. S42 of van der Vusse's paper were apparently reasonable, but said that the one in which he was particularly interested was the first, *i.e.* keeping the Reynolds number constant. He was not sure that it was generally appreciated that if the Reynolds number were kept constant it meant that the actual power input on the larger scale was smaller than the power input on the smaller scale—not merely the power input per unit volume, but the actual power input.

If the Reynolds number was constant, nL^2 was constant. With turbulent mixing the power was proportional to $\rho n^3 L^5$, which meant that $P \propto \rho(nL^2)^3/L$. This meant that the actual power on the larger scale was less than the power on the smaller scale. That was obviously not impossible, but it was highly unlikely. It meant that in practice keeping the Reynolds number constant was not a good basis. The same argument applied to mixing in the laminar region.

He had one other comment for which he had no experimental evidence. Keeping the tip-speed constant in scaling-up was a very well known criterion and worked in many instances, but it was worth remembering that when scaling-up at constant tip-speed the r.p.m. obviously dropped, so that there was a strong case when scaling-up to increase the number of blades on a paddle, or in other words to depart from geometrical similarity. One then had both a constant tip-speed and also a constant " rate of beating ", *i.e.* the number of blades passing any given point in a given time could also be kept constant.

Mr. J. HARDEBOL said that from Mr. de Gelder's paper on cyclones it would be understood that the separation of the particle depended on the diameter of its equilibrium orbit with regard to the diameter of the overflow aperture, the diameter of the orbit being determined by the forces acting in a radial direction, the forces being the centrifugal force and the frictional force exerted by the radial flow. It was also understood that the criterion for the separating performance of the cyclone was obtained by balancing these two forces, and as a consequence equation (3) on p. S50 probably described equilibrium conditions, which meant that the conditions in the cyclone were supposed to be stationary with respect to the radial position of the particles.

Opposing this line of thought was the idea that in view of the rather short residence time of the particles, an equilibrium or stationary orbit would not be established, and that in such cyclones non-stationary conditions would prevail. In that case the criterion for separation could be obtained from the condition that during its residence time a particle had to travel from its initial position at the inlet cross-section to a position near the apex just outside the core in which axial up-flow occurred, assuming that a particle caught by the upflow would not be separated.

Before choosing any correlation between separation performance, fluid properties, and cyclone dimensions, the underlying mechanism of separation had to be established and agreed upon. As long as this had not been done, it was best to proceed along both lines of thought and find how both the resulting expressions compared with the experimental results. He would therefore appreciate Mr. de Gelder's comments on the comparison of stationary and non-stationary conditions in cyclones, or perhaps some information in addition to that already given in the paper, stating the reasons for preferring the stationary approach to the non-stationary one.

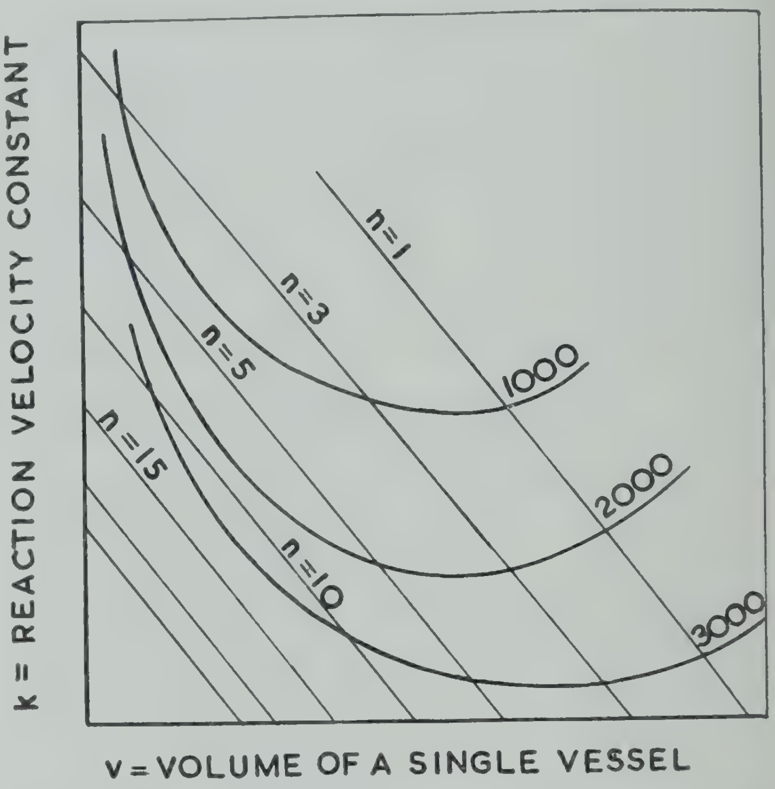
Mr. C. J. STAIRMAND in a written contribution read at the meeting said :

Mr. de Gelder's mathematical analysis of the mechanism of operation of a cyclone was of great interest. It was, however, confined to a study of the flow of the gas or carrying fluid within the cyclone and took no account of the secondary effects such as particle bouncing, or re-entrainment from that point in the cyclone at which the dust was discharged. As he had shown earlier, these effects might lead to a very considerable deterioration in performance and are thus very important. He had used Mr. de Gelder's formulae to predict the pressure drop for a number of cyclones for which we have experimental figures, and it appeared that his formulae only gave the correct result when the cyclones were of conventional shape. Any departure from the normal cylinder-cum-cone design led to wide departures from the predicted figure. In his own analysis of cyclone flow pattern,[11] he considered the vortex within the cyclone as being controlled by the energy applied via the inlet, and the frictional force on the wall of the cyclone. In almost all cases, and particularly for cyclones of rather special shape, this analysis gives considerably better agreement with the observed values than Dr. de Gelder's formulae. An important part of his own method of calculation was that it enabled one to determine the ratio between the actual velocity in the inlet duct and the velocity in the vortex at the point where the inlet gas joins it. He had found by observation in transparent models, confirmed by experience, that an inlet velocity some 5–10% higher than the spinning speed at the inlet radius gave the least disturbance to the vortex, thus enabling the correct inlet design to be determined. The actual geometry of the cyclone was a matter for experiment, and he had in a later paper given specifications for what he considered to be the best design.[12] It was of interest to know that these designs do, in fact, agree with the strictures made by Dr. de Gelder.

Another point of interest was that the denominator in Mr. de Gelder's equation (2) had a value of $0 \cdot 94 \pm 0 \cdot 05$ for a very wide range of cyclone designs. This suggested that the behaviour of normal gases within the cyclone approaches very closely that of the ideal fluid and made one wonder whether the correction for departures for α_∞ was worth while.

Dr. K. SCHOENEMANN said that at the Technische Hochschule, Darmstadt, they had developed a method for the investigation and design of new chemical processes which combined the aspects of economy stressed by Mr. Dickerson, the choice between number and size of reaction vessels as discussed by Prof. Denbigh, and the further factors involved as outlined by Dr. van de Vusse with respect to slurry reactors.

If with a slurry reaction, *e.g.* the hydrogenation of sugar into sorbitol, it was necessary to obtain a nearly theoretical yield because the desired product could not be separated from the raw materials, Dr. Schoenemann said that they started with bench or small-scale experiments with good mixing. From such experiments the reaction velocity constant could be estimated as a function of pressure and catalyst concentration. If then, for the desired degree of conversion, the investment cost of a cascade of reactors was plotted as a function of both the number (inclined parameter lines) and the volume of the reaction vessels (abscissa), and additionally of the reaction velocity constant (ordinate) for each value of velocity constant, there would be a distinct combination of volume and number of the reaction vessels, which would represent the minimum investment cost. If the operating costs were also considered, the curves would be slightly modified, but would always show distinct minima (Fig. 1). Thus the whole development work could be rationalised by predicting whether it would really be economic to reach a higher velocity constant, for example by use of higher pressure and higher catalyst concentration. The detailed development work could then be limited to those points on the graph representing conditions which were economically promising.



n = number of reaction vessels in series
curves = curves of equal total costs of the desired reaction unit at a desired degree of conversion

Fig. 1.—*Investment costs for a cascade of reactors*

Dr. Schoenemann mentioned that his co-authors Dr. Hoffmann and Mr. Bill had been carrying out systematic work on this basis which they hoped to publish shortly.

Mr. TH. ZWIETERING wrote :

Dr. van de Vusse has considered various requirements which must be fulfilled for obtaining a sufficient similarity between model and technical apparatus.

One important condition is that all the particles of the dispersed phase must be in suspension. If this can be realised both in the model and in the full-scale vessel, the rate of heat and mass transfer from the fluid to the dispersed particles will be the same.

Because this operation, the stirring of slurries, is frequently encountered in the chemical industry, and quantitative data for the required stirrer speed cannot, in most cases, be found from the literature, a rather extensive investigation has been carried out in the Central Laboratory of Staatsmijnnen (Dutch State Mines).

The results of this investigation have shown that in the case in hand the rule of equal power per unit volume is a safe rule for scaling-up. However, it is not exact, for with all stirrers which we have used, we found that the required power is not proportional to L^3, but to $L^{2\cdot5}$, which means that the power per unit volume can be decreased with $L^{-0\cdot5}$. Therefore, the rule as mentioned by Dr. van de Vusse is not valid for " scaling-down ".

A second result was a confirmation of the finding of Kneule that a propeller stirrer is in general very much more efficient for this operation than paddles or turbines.

Another point that may be worth mentioning is the possibility of scaling-up without maintaining an exact geometrical similarity. When designing the full-scale apparatus, it is often convenient to use one of certain fixed stirrer speeds, especially for high speed stirrers, which run at the same speed as the motor. In this case one could fix the stirrer speed at a value which is not too much different from that which follows from the scaling-up rule, and then change the ratio of stirrer diameter to vessel diameter so as to maintain the same power. This procedure is only safe when using propellers, but for paddles and turbine stirrers the required power depends strongly on this geometrical ratio. For these radial stirrers the required power increases with decreasing ratio of stirrer diameter to vessel diameter. Therefore it is advantageous for these stirrers to use a rather large stirrer with a corresponding low speed.

It is hoped to publish the results of these experiments in the near future.

Authors' Replies

Dr. G. H. Reman said that Dr. Pratt had criticised him for the use of dimensional instead of dimensionless groups. He wondered whether this criticism was serious. It was easy to give an example to show that there were certain cases where the same technique could be applied. In the case of flow of fluid through a pipe, a dimensionless correlation exists between $\frac{\Delta p}{\rho v^2}$, and the Reynolds number, which was $V\rho d/\mu$. If one had to design a pipe for a certain flow and did not know anything about the properties of the liquid, then in principle—and even without measuring these properties—it would still be possible to design the pipe by carrying out a model experiment by taking a pipe and measuring the pressure difference as a function of the velocity of flow and plotting $\frac{\Delta p}{l}$ against vd. This relation could be used for the perfect design of the large-scale pipe.

He thought that, in principle, the method he had used for designing commercial contactors was the same as that outlined for the pipe flow example. He could indicate what dimensionless groups were involved. For instance, for the capacity correlation, the properties of the liquid system entered the correlation as the velocity had to be made dimensionless by introducing the density difference, $\Delta\rho$, and the constant of the gravitational acceleration g, the viscosity of the continuous phase η_c and the density of the continuous phase ρ_c, would be related as follows :

$$\frac{V}{\left(\frac{\Delta\rho g\eta_c}{\rho_c^2}\right)^{1/3}}$$

The other term in the capacity correlation concerned the energy per unit volume, E, and would appear :

$$\frac{\eta_c{}^5 E}{\sigma^4\rho_c}$$

One might try to correlate capacity data with either of the above-mentioned groups, but in fact the energy group was used ; another group, which involved only the properties of the liquid had to be added, and that made it very complicated.

They preferred, therefore, to design in the way mentioned. Liquid-liquid systems were always difficult. Experimental determination in a laboratory contactor of the design relation between the velocity or capacity and a quantity proportional to the power input per unit volume was much more reliable. However, it had to be stressed that a design method as indicated in the paper required a good knowledge of the dimensionless groups involved.

The comment was made regarding Fig. 2 that points were shown where there was no flooding. It was notoriously difficult to obtain exact flooding data with liquid-liquid extraction columns. All that one knew was that at a certain moment the column was not flooding, and then one changed the conditions a little and found that the column was flooding. For that reason he had given both non-flooding points and flooding points, and the curve must be somewhere in between. The way in which they designed these contactors was to take the design capacity equal to 65% of flooding capacity. That provided a margin in operation, and this margin was really essential in case of practical extractions.

Answering the question of whether one could expect drop size and mass transfer coefficients to depend on power input Dr. Reman replied that in the literature there were a great many data which indicated that drop size, at least in stirred vessels, could be correlated by means of power input. So far as the mass transfer coefficient was concerned there was still a division of opinion. Many people said that it could be done, while others held that other correlating variables should be taken. His view was that it was merely a question of personal preference, and that the way in which the efficiencies could be correlated by means of power input at least indicated that the errors would not be serious.

He had been asked why in Fig. 9 (p. S539), no attempt had been made to bring the curves together by including in the correlation the factor of the square root of the extraction coefficient. That could have been done, but, if it were done, it would have also been necessary to account for the change in hold-up which occurred because of the change in phase ratio; and, since these hold-ups were not known, it was impossible to improve on the correlation in that specific case by bringing in the square root of the extraction factor.

Dr. Pratt asked for the maximum diameter of column to which they could scale-up at present. They had a column operating with a diameter of $2\frac{1}{2}$ m., and there were designs for columns with a diameter of $3\frac{1}{2}$ m., although those columns were not yet in operation. Dr. Pratt had also asked about the number of stages to which they would go, and referred to 10–15 theoretical stages. There were cases where it was quite easy to get 15 stages. In general the maximum total height of the contactor could be taken to be about 15 m. In general less than 1 m. was needed for a theoretical stage.

Dr. Thornton asked a question about Fig. 3, showing the influence of phase ratio on maximum capacity. Dr. Reman mentioned that this relation had been obtained in developing a theory for the spray extraction column.[13] That theory started from the fluidisation of solids with a liquid. Further, they took into account the relation for free-falling velocity of drops and a Weber number relation for the size of the drops. By doing that they derived an equation which made it possible to predict hold-ups. The hold-ups predicted corresponded very well with the experimental ones for spray columns. The relation shown in Fig. 3 could also be calculated by that theory, and they had checked it for the rotating disc contactor.

Dr. Thornton had also commented on Fig. 5. It was stated in the paper that the decrease in efficiency with increasing rotor speed was due to back-mixing. That this was the case could be concluded from the fact that when the height of the mass transfer stage in the apparatus was compared with the height of the mixing stage it was found that the height of the mass transfer stage and the height of the mixing stage were of the same order of magnitude. The drop sizes in rotating disc contactors were always of the order of at least 1 mm. and in most cases 2 mm.

Mr. Eijsberg wondered whether there was a contradiction between the example of Fig. 9 and the correlation given in equation (3), and mentioned that in general this correlation was true only for the quantity $(N^3R^5)/(HD^2)$ for values less than about 0·1. In this case they had a very difficult system, so that the height of the mass transfer stage was still large compared with the height of the mixing stage. This meant that the mixing in this case had very little effect; but in another system with easy mass transfer there would have been a very great difference in this region of power inputs.

Equation (3) had been derived for the case where there was a spread in residence time and also for the case where there was no spread in residence time of the continuous and dispersed phases and the formulae were almost exactly the same.

Dr. J. G. van de Vusse, replying to Mr. Willemse's question regarding the border between isotropic and non-isotropic turbulence, said that it was more or less the same as going from the concept of the mixing stage to the concept of the diffusivity constant. There was a relation between the number of mixing stages, $N = \frac{1}{2}\frac{vL}{E}$, where v was the velocity, L the length, and E the diffusivity; and this could be used if the number of stages was about 5–10. This was more or less the border between one concept and the other. He did not know, however, that it was possible to apply the concept of pumping capacity which was used in the mixing of miscible liquids to the case of slurry reactors. He felt that with slurry reactors it was necessary to deal with velocity and not with velocity × sectional area, which was the pumping capacity. That was the reason why on the basis of the power consumption much better correlations were obtained than in terms of pumping capacity. The propeller stirrer had a much lower power consumption for maintaining suspension than the paddle stirrer, and for the same power consumption, the velocity in the propeller was much higher and gave better suspension.

It would be very nice if it were possible to correlate all the processes where mixers and stirrers were involved on one basis. For the case of isotropic turbulence that was a relation between mixing time T and diffusivity E in this sense, that the diffusivity could be related to the height of the mixer H over the mixing time, $E = \frac{H^2}{T}$. He had no experience of vibrational mixers or jet mixers.

He agreed with what Mr. D. C. F. Pratt had said about the Reynolds number. Hixson in his experiments with mass transfer and heat transfer correlated against Reynolds number and obtained a good correlation, so that there might be applications where the Reynolds number could be used; but in general it was better to use power consumption or tip velocity, depending on the process.

Dr. de Vusse was glad that Prof. Schoenemann could use this theoretical and experimental evidence for his economic valuation. It was always nice to hear that there was a practical use for the work which one was doing.

Dr. A. L. DE GELDER said that he introduced the Froude number as criterion in scaling-up problems because this number indicated the number of times the separatory forces of a cyclone were greater than those of a settling tank. Using this number, the settling tank and the cyclones might be arranged in the order of increasing separating effect. Scaling-up would then be aimed at obtaining a cyclone that might be assigned to the same place in that series as was occupied by the selected model cyclone.

A fundamental outline of the backgrounds of this problem was given on p. S. 47.

The value of the formulae had been examined experimentally; by way of example one result was shown in Fig. 1 (p. S50). The small circles denoted measured values; the drawn line had been calculated. The calculation was carried out without making use of empirical results.

In reply to Mr. Bradley, Dr. de Gelder reiterated that no empirical constants nor data had been incorporated in his formulae. They were valid both for hydrocyclones and for gas cyclones provided that in the former no cavitation occurred and that the compressibility in gas cyclones was not considered. The formulae would therefore hold for incompressible "fluids with viscosity" on condition that cavitation phenomena were absent.

In connection with Mr. Stairmand's remarks Dr. de Gelder said that the calculation was carried out for conventional cyclones by which he understood cyclones of the model suggested by Ter Linden. However, this explanation was also valid for other models as would be shown in future publications.

Correction for the viscosity might indeed be neglected for dust cyclones under normal conditions; this left only the term α_∞. Under the above conditions, however, the formulae were also valid for liquid cyclones where the viscosity might be of importance. Using the Reynolds number it was then possible to determine the conditions under which a cyclone might be used and to form an estimate of the energy requirements.

Mr. B. F. WARNER, said that Dr. H. R. C. Pratt's approach to scaling-up mixer-settlers was a valuable one. Essentially, this was to determine the fundamental theory of the volume required to effect a given amount of transfer in a system and also the volume required to get a given degree of settling; but the problem with which Mr. Warner's paper was concerned was that of scaling-up when fundamental data were not available. He had tried to indicate in the paper that if the geometrical scale factors and the standard method for power input were used it was possible to scale-up a mixer-settler from the laboratory size to one processing several tons/hour. In fact this had been done.

With further reference to Dr. H. R. C. Pratt's contribution, it was always necessary to make some allowance for the presence of interfacial solids affecting the settling rates, and these, so far as Mr. Warner knew, were not amenable to scaling-up procedures. Where there were no solids, it was possible to get a very reasonable approximation by scaling-up geometrically.

On the question of the mixer itself, Prof. Denbigh in the first session had made Mr. Warner's point very well by pointing out that if the residence time was long compared with the reaction time the design of the mixer was less important. That stated very well what had been found at Windscale. Since they handled radioactive materials, they had gone from mechanical stirrers to completely different types of mixing, using pneumatic stirrers, so that they would have to start all over again.

Dr. Thornton's points were very valid, but this was the first time that the workers at Windscale had been called pessimistic; Mr. Warner suggested that " cautious " might be a more appropriate word. In the scaling-up of columns, if one had to determine the empirical factors which Dr. Pratt had developed and to which reference was made in the paper, the shielding factor X_m and the longitudinal mixing factor F_m, this had to be done on a column of approximately the same diameter and packing, and one would do what they themselves had done in 1949. One would now know a good deal more about the relationship between the different factors in the column, but the process would be much the same. Mr. Warner would say, as a personal opinion, that it was necessary to be very cautious before attempting to scale-up from a 2 in. to a 12 in. column by the methods which they had outlined. He would not risk it for any plant which was to be surrounded by concrete and which could not be altered once the process was started.

Dr. Lessing had made some most interesting comments. The effect of the downcomers was undoubtedly primarily to improve the flow pattern of the liquors in the column and to remove channelling. The fact that they did not reach the same efficiency as with the 2 in. column —and it should be emphasised that it was not a theoretical figure—in the 12 in column was explained largely by Dr. Pratt's work, where the importance of the free path of the droplet in the packing was clearly demonstrated. When working with a 2 in. column, with say, $\frac{1}{8}$ in. packing, and droplets of 2 mm., the free path was much smaller than in the larger packing, and the characteristic velocity was different for that reason.

Mr. Warner agreed with Dr. Lessing that wetting made a very big difference in practice. They did some work many years ago on graphite rings in which their solvent did wet the packing, and their results had been adversely influenced.

Dr. Reman's comments had been very interesting, and Mr. Warner wished to thank Dr. Klinkenberg for a far more eloquent reply then he himself could have given; but Dr. Reman's point had been of value, because it drew attention to the fact that there was another relation than the one given in the paper for the relationship between H.T.U. and H.E.T.S. and showed how H.T.U. could be converted to an actual column height. With regard to Dr. Reman's remarks about the rotary column, they had done some work on this type of contactor but the remarks in the paper referred entirely to the selection of micro-contactors for developing the chemical process. That was where the difficulty of taking accurate samples stage by stage in micro-contactors arose. They had considered the use of full-scale rotating disc contactors and had done a small amount of development work at Windscale, but a great deal more had been done by Dr. Thornton at Harwell. The reason they had not been chosen was that rotating machinery in highly radioactive plants was something which as yet they were not prepared to recommend.

The remarks about interfacial area as a parameter for the settler might interest Mr. Warner's colleague Mr. Williams, who had done some work in this field. They had not been aware of the existence of the Shell paper and were grateful to Dr. Reman for drawing their attention to it. In their limited experience, using rather long settlers, where they had critical assemblies of fissile material, they found that the shape of the settler had quite as much effect as any other factor on the settling pattern. In other words, a long, thin settler was sometimes better than a short, fat one. Probably Dr. Reman knew that very well.

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THE PERFORMANCE OF TECHNICAL APPARATUS FOR GAS ABSORPTION

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SUMMARY

Scaling-up problems in gas absorption are illustrated by a discussion of the packed column. Dimensional analysis shows that undistorted models cannot be obtained if the diameter of the packing particles is changed. In accordance with this, most of the experimental data on gas absorption published recently were obtained with full-size packing materials.

One scaling-up procedure is to change the overall dimensions of the absorption apparatus. This affects the liquid distribution over the packing which may have an important influence on the absorption effect.

These considerations can be generalised by distinguishing between the elements which should not be changed in size during the experiments and the effect on distribution of the over-all dimensions of the apparatus.

By way of example the absorption of carbon dioxide by water in a packed column is discussed. For this process laboratory data as well as descriptions of semi-technical and technical experiments can be found in literature.

Introduction

In considering the use of small-scale experiments as a means of predicting the performance of technical gas-absorption apparatus, the evolution of general rules is impracticable as the different types of apparatus are so numerous that this would require an almost complete discussion of scaling-up problems as they affect chemical engineering. Besides, there is no reason for assuming that the scaling-up rules for gas absorption processes differ from the rules applying to other unit operations.

However, certain definite types of apparatus are widely employed for gas absorption and the data obtained may be used for evolving scaling-up rules for these particular types. For this reason absorption in a packed column will first be discussed. The discussion will be limited to the absorption of carbon dioxide in water, a process in which the absorption rate is determined by the rate of the physical mass transfer in the liquid phase. Subsequently the procedure will be examined to see to what extent the results can be generalised.

Liquid Flow

Flow Over the Packing

If a comparison is to be made between two irrigated packed beds, the two necessary conditions essential for similar mass transfer processes are: geometrical similarity of the beds and similarity of flow of the liquid over the packing. The influence of the walls of the apparatus on these factors is for the present disregarded.

Geometrical similarity presupposes that the individual packing bodies and the way in which they are packed should be similar.

The conditions of similarity for the flow of a liquid over a packed bed have been discussed by Rushton.[1] It is assumed that the flow is not influenced by the presence of some other phase, and that the influence of surface tension is disregarded.

The following quantities are then left for consideration :—

u = linear velocity,
D = diameter of the packing bodies,
ρ = density of the liquid,
μ = viscosity of the liquid, and
g = acceleration due to gravity.

These quantities may be combined into two dimensionless groups :—

$$\frac{uD\rho}{\mu} = Re = \text{Reynolds number.}$$

$$\frac{Dg}{u^2} = Fr = \text{Froude number.}$$

The flow over the two beds is similar if both Re and Fr are equal. If subscripts 1 and 2 are used for denoting the quantities pertaining to the two beds

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* Staatsmijnen, in Limburg, Central Laboratory, Geleen.

$$\frac{u_1 D_1 \rho_1}{\mu_1} = \frac{u_2 D_2 \rho_2}{\mu_2} \text{ and } \frac{D_1 g}{u_1^2} = \frac{D_2 g}{u_2^2}$$

from which it follows that :

$$\frac{\mu_1 \rho_2}{\mu_2 \rho_1} = (D_1/D_2)^{3/2}$$

Since for most liquids ρ is about equal to 1 g./c.c. :

$$\mu_1/\mu_2 \approx (D_1/D_2)^{3/2} \quad . \quad . \quad (1)$$

It is often impossible to satisfy this requirement. If, for instance, in a technical apparatus 1 in. rings were irrigated with water, model experiments using $\frac{1}{5}$ in. rings would require the use of a liquid having a viscosity of 0·09 centipoise, which is practically impossible.

The requirements of similarity are still more difficult to satisfy if the surface tension σ is taken into account. This leads to the requirement that :

$$\frac{\sigma_1}{g \rho_1 D^2{}_1} = \frac{\sigma_2}{g \rho_2 D^2{}_2}$$

which, if ρ remains unchanged, implies that :

$$\frac{\sigma_1}{\sigma_2} = \left(\frac{D_1}{D_2}\right)^2 \quad . \quad . \quad (2)$$

Although in some cases it may be possible to satisfy requirement (1), it is very unlikely that a model liquid will be found which satisfies both requirements (1) and (2).

It is also clear from visual observation that the influence of the surface tension on the irrigation of the packing cannot be neglected. If a bed of Raschig rings of $\frac{1}{5}$ in. diameter is irrigated with water, the space between the rings is in many places completely filled by the liquid. When the same process is carried out with technical-scale rings, there is only a small accumulation of water around the points of contact between the rings.

It follows that it is practically impossible to scale-up an irrigated packed bed. Therefore, absorption experiments should be carried out with exactly the same material as will be used for the technical-scale process. Accordingly, nearly all recently published experimental data on absorption processes carried out in packed columns were obtained by the use of technical-scale packing.

Liquid Distribution in an Irrigated Packed Column

To what extent may smaller-scale columns be used for these experiments ? The minimum diameter of the experimental column is determined by the requirement that the arrangement of the rings shall not differ widely from that in the technical-scale column. A deviation of the arrangement near the wall has always to be considered. Therefore, there is a certain minimum value of the ratio between the column diameter D_c and the diameter of the packing bodies D. Empirically determined minimum values of D_c/D can be found in literature ;[2, 3] it is there stated that D_c/D should be greater than 10. When 1 in. rings are used the experimental column should be given semi-technical dimensions.

It would be reasonable to expect the absorption rate in the technical column to have the same value as the rate in the experimental column if in both cases the liquid were perfectly uniformly distributed over the packing material and if this distribution remained uniform as the liquid flowed down. An even distribution of the liquid in the top of the column is not always obtained and the arrangement of packing material introduced at random into a column always shows irregularities. As a result, there will be poorly irrigated parts where the liquid is only slowly replaced and becomes saturated with the component to be absorbed. On the other hand, there will be places where channelling occurs, *i.e.* where the liquid flows past in a stream. Also part of the liquid will flow off towards the wall. Liquid which has once reached the wall will hardly ever return into the packing (wall effect).

The streams of liquid issuing from the bed differ because of these effects in their degree of saturation with the substance to be absorbed. Mixing of these streams produces the final liquid. The total absorption efficiency thus becomes dependent on the liquid distribution.

In order to forecast the performance of a technical apparatus on the basis of experimental data with any degree of certainty, it is essential to have an idea of the liquid distribution in the two columns.

The literature on liquid distribution in a packed column relates almost exclusively to the radial distribution, which depends not only on the performance of the distributor situated over the packing material, but also on the wall effect.

In the experiments carried out in the Central

Laboratory, special attention was paid to the distribution of the liquid among the individual packing bodies. A number of packing bodies were constructed as measuring cells and by measurement of the electrical resistance it was possible to determine the thickness of the liquid layer passing over them. Each of these cells could be placed at any desired spot in the packed column. It was found that the variation in the irrigation of individual packing bodies lying at equal distances from the axis in the same horizontal cross-sectional plane is much greater than the differences between the mean values of the irrigation for distinct concentric rings. This investigation has not yet been concluded.

The ultimate aim of these investigations is to determine the conditions of the liquid distribution in a technical-scale column which correspond sufficiently to those occurring in an experimental column to enable the performance of the technical column to be forecast by means of experimental data. As long as such data are lacking, there is always the possibility that the results obtained from a technical apparatus will differ from those from an experimental column because of the differences in liquid distribution.

General

The same reasoning is, in principle, applicable to processes governed by resistance in the gas phase. The flow of gas through a ring-packed column will differ according to whether or not the openings between the rings are filled with liquid. However, the influence of the irrigation of the packing material on the absorption effect will be smaller than with transport processes in the liquid phase. Moreover, the way in which the gas is distributed may also exert an influence.

Previously we distinguished between the irrigation of individual packing bodies and the distribution of the liquid over the bed. In general we may distinguish :

(1) the elements of the apparatus (the individual packing bodies),

(2) the arrangement of these bodies (the way in which the packing material is accumulated), and

(3) the way in which gas and liquid are distributed through this arrangement.

It was found that the size and shape of the elements and their arrangement had to be the same in the experimental and technical columns. Therefore, scaling-up is restricted to the overall dimensions of the apparatus. The degree to which scaling-up may be possible is determined by its effect on the distribution of liquid and gas.

For other types of apparatus in which gas and liquid are brought into contact the same considerations hold as for the packed column. In a bubble cap column, for instance, the formation of bubbles on the caps is determined by gravity, viscosity forces, and surface tension. In model experiments, bubble caps of the same dimensions as used in the technical apparatus have to be employed, while the arrangement of the caps on the plates should also be the same. The flow of the liquid over the plate in the experimental column may, however, differ from the flow in the technical apparatus. In technical constructions special devices are often used to influence the flow of the liquid. Finally, in columns of large diameter an uneven distribution of the gas may occur, owing to the loss of head of the liquid in flowing over the plate.

In a recent article[4] mention was made of two technical bubble-cap columns, designed on the basis of laboratory experiments. The model comprised a part of one plate with technical-scale bubble caps. The large columns were 3 ft. in diameter and each had 50 plates. The mean plate efficiency corresponded reasonably well with the value predicted from the laboratory experiments.

It will be clear that the general considerations given above do not apply to all types of absorption apparatus. We may conclude that it is impossible to give a simple scaling-up rule for gas absorption apparatus in general or even for one particular type of apparatus. In this connection we may refer to an enquiry[5] in which several experts in chemical engineering design were asked what, in their opinion, was the maximum scaling-up ratio for certain types of apparatus. As long as this question is not further defined, the results of such an enquiry have little value. This is clearly seen from the answers received : for packed columns the scaling-up ratios given varied from 1 to 100.

Example

The absorption of carbon dioxide by water in a packed column

A suitable example to show the influence of the dimensions of the apparatus on the absorption efficiency is provided by the absorption of carbon dioxide in water, carried out in a packed column. Results of laboratory experiments as well as measurements on semi-technical and technical columns can be found in literature.

A measure of the absorption efficiency is the quantity $k_L a$, the coefficient of mass transfer in the liquid per unit of volume of the column. In practice, $k_L a$ proves to depend mainly on the superficial liquid velocity u. Dimensional analysis shows that in similar situations the influences of various factors on k_L may be expressed as a relationship between the following dimensionless groups :

$$\frac{uD\rho}{\mu};\ \frac{Dg}{u^2};\ \frac{\sigma}{g\rho D^2};\ \frac{k_L D}{D_L};\ \frac{\mu}{\rho D_L}.$$

In order to express the relation between $k_L a$ and u, van Krevelen and van Hooren[6] used an equation which may be simplified to :

$$\frac{k_L a}{a^2_p D_L} = c\left(\frac{u\rho}{a_p\mu}\right)^{0.8}\left(\frac{\mu}{\rho D_L}\right)^{0.4} \quad . \qquad (3)$$

where the diameter of the packing bodies, D, has been replaced by the reciprocal value of a_p, the total surface area per unit volume of the packing material. The experiments carried out by Sherwood and Holloway[7] on the desorption of oxygen from water in a packed column proved to be well covered by equation (3).

In Fig. 1 the group

$$\frac{k_L a}{a_p{}^2 D_L\left(\frac{\mu}{\rho D_L}\right)^{0.4}}$$

has been plotted logarithmically against :

$$\frac{u\rho}{a_p\mu}.$$

Series of laboratory experiments and semi-technical measurements on the absorption of carbon dioxide in water can be graphically represented by a straight line. For the sake of clarity the experimental points have been omitted in the graph. The results of the technical-scale experiments showed a wide spread, so that in Fig. 1 only an area could be indicated.

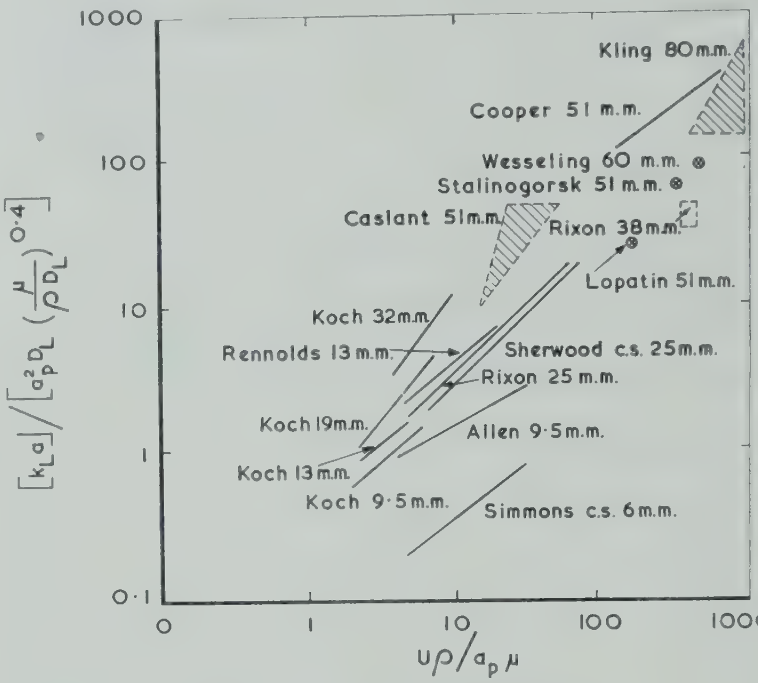

Fig. 1.—*Application of equation (3)*

From the figure it is seen that the dimensions of the packing material exert a great influence on the absorption efficiency. This is completely in agreement with expectations based on the foregoing considerations. The results of measurements carried out with the same packing material in different types of apparatus cannot be represented as lying on one line, although in this case the deviations are smaller. This is also seen in Table I, where the values of constant c in equation (3) are given for the various series of tests.

TABLE I.—*Values of the Coefficient C*

Authors	Ref.	Diam. column (m.)	Diam. rings (mm.)	Number expts.	c mean	c range
Simmons *et al.*	7	0·09	6	11	0·06	0·05–0·11
Koch *et al.*	8	0·15	9·5	13	0·26	0·18–0·30
Allen	9	0·05	9·5	10	0·25	0·19–0·34
Koch *et al.*	8	0·25	13	11	0·38	0·32–0·44
Rennolds	10	0·08	13	20	0·66	0·57–0·74
Koch *et al.*	8	0·25	19	24	0·56	0·40–0·76
Rixon	11	0·38	25	26	0·84	0·50–1·03
Sherwood *et al.*	12	0·25	25	17	0·63	0·52–0·73
Koch *et al.*	8	0·25	32	24	1·10	0·85–1·34
Cooper *et al.*	13	0·76	50	31	2·38	2·04–2·73
Rixon	11	0·69	38	12	0·32	0·19–0·39
Kling	14	2·4	50	2	0·74	0·64–0·84
Lopatin	15	2·4	50	2	0·45	0·44–0·46
Caslant	16	2·3	50	16	2·15	1·23–3·09
Wesseling	17	2·0	60	2	0·72	0·72–0·72
Kling	14	1·4	80	7	1·35	0·72–2·35

In considering the figures it should be remembered that the poor reproducibility of the measurements is not the only cause of the spread of the c values, as c was calculated by means of a power exponent of 0·8 for the Reynolds number, while the lines in the figure all have different slopes. Besides, in a number of experiments loading occurred to a certain extent. As a result, the absorption efficiency became dependent also on the gas rate.

Conclusion

The final conclusion is that for this process, in some cases the absorption effect in a technical column amounts only 25% of that predicted on the basis of semi-technical experiments. A more accurate prediction is not possible as long as further data on the liquid distribution in both cases is lacking.

Symbols Used

a = active surface per unit volume (m.$^{-1}$).
a_p = total surface per unit volume (m.$^{-1}$).
c = constant.
D = diameter of packings (m.).
D_c = diameter of column (m.).
D_L = diffusivity in liquid (m^2. sec.$^{-1}$).
g = acceleration of gravity (m. sec.$^{-2}$).
k_L = liquid mass transfer coefficient (m. sec.$^{-1}$).
u = velocity (m. sec.$^{-1}$).
μ = absolute viscosity of liquid (kg. m.$^{-1}$ sec.$^{-1}$).
ρ = density of liquid (kg. m.$^{-3}$).
σ = surface tension (kg. sec.$^{-2}$).

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SCALE UP EXPERIENCE IN THE REMOVAL OF HYDROGEN SULPHIDE FROM EXHAUST GASES FROM A VISCOSE FACTORY

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SUMMARY

The very large volumes of air to be treated and the low concentrations of hydrogen sulphide created special problems in this project. Pressure losses had to be minimised and costs kept low, particularly since the process is mainly carried out to avoid atmospheric pollution, rather than for sulphur recovery.

Courtaulds use the "Ferrox" process, wet scrubbing with an alkaline suspension of hydrated ferric oxide. The paper briefly describes the laboratory work which was carried out to elucidate the chemistry of the process, and outlines the pilot plant work on a 2 ft. square scrubbing tower. The reasons are given for adopting, at that stage, vertical spray-type towers. Experience with the prototype full-scale vertical tower is described, and running data are given.

Reasons are given for changing to a horizontal scrubbing unit for the next development, and the present horizontal unit is described and performance figures given.

The paper discusses the difficulties of defining the performance of these units in terms of a design coefficient, stating the type of questions which arose and the answers obtained.

Introduction

Most of the sulphur content of the carbon disulphide fed into a viscose rayon factory passes up the chimney in the exhaust air as carbon disulphide, hydrogen sulphide, and other sulphur compounds. The quantity of this foul air is usually very large and in the factory to which this paper refers, a single hydrogen sulphide scrubbing plant would have to handle a total flow of 250 000 cu. ft. of air per min., saturated with water vapour and containing 800–1000 p.p.m. (by volume) of carbon disulphide and 300 p.p.m. of hydrogen sulphide plus smaller concentrations of other sulphur compounds.

The hydrogen sulphide should be removed from this stream (down to 10–20 p.p.m.) mainly to avoid atmospheric contamination, but its removal would also be desirable for any future carbon disulphide recovery process. The economic aspects of sulphur recovery from hydrogen sulphide are not very significant.

Full-scale plants operating on the "Ferrox" process had already been operated to some extent by Courtaulds, but they had not functioned satisfactorily. It was therefore decided to reconsider the problem, with particular reference to a staple fibre factory, starting with laboratory work on the chemistry of the process, followed by pilot-plant work and full-scale design. Alternatives to the Ferrox process were considered, but the advantages and disadvantages of these are not relevant to this paper.

This paper is not put forward as an example of a scale-up based on academic chemical engineering principles, but rather as an example of how scaling-up often proceeds.

Chemical Aspects of the Process

Ferrox is a "wet" process in which hydrogen sulphide is absorbed in an alkaline liquor containing a suspension of hydrated ferric oxide, referred to as the catalyst. The main overall reaction is :—

$$2\,H_2S + O_2 \longrightarrow 2H_2O + 2S$$

but there are many side-reactions.

In the process operated by Courtaulds, the hydrogen sulphide is absorbed in a spray chamber from which the spent liquor passes to a tank where most of the oxidation occurs under the influence of the catalyst. Air is bubbled through this tank to provide the oxygen required, and also to maintain the catalyst in suspension and cause the elemental sulphur which forms to float to the surface.

The alkalinity of the liquor is due to sodium carbonate and bicarbonate. The liquor also contains thiosulphate, sulphate, polysulphides, sulphite, and other sulphur compounds, all formed as by-products.

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The absorption of hydrogen sulphide results from its solution and subsequent rapid reaction with the sodium carbonate :—

$$Na_2CO_3 + H_2S \rightleftharpoons NaHS + NaHCO_3 \; . \qquad (1)$$

The equilibrium normally lies towards the right-hand side. Since equation (1) represents an equilibrium there must be a partial pressure of hydrogen sulphide above the surface, related to the free hydrogen sulphide in solution. This partial pressure will depend not only on equation (1) but on the other salts in solution which reduce the solubility of hydrogen sulphide in water. Any partial pressure of hydrogen sulphide at the interface arising from sulphides in the liquid obviously retards the absorption.

Laboratory Experiments

Table I gives data obtained in a 1 in. diam. falling film column, together with the corresponding liquor analyses. Gas-film resistance is considered to be the limiting factor in the absorption of hydrogen sulphide by caustic soda solutions since the reaction

$$2NaOH + H_2S \longrightarrow Na_2S + 2H_2O$$

is very rapid and lies wholly to the right owing to the excess of caustic soda present in the experiments. Compared with caustic soda, carbonate solutions give a lower overall coefficient which is further reduced by dissolved salts. The lowering is due to the effect of the equilibrium represented by equation (1) and the resulting interfacial pressure of hydrogen sulphide, which is not negligible compared with the low partial pressure of hydrogen sulphide in the outlet air stream. Equilibrium (1) suggests that sodium bicarbonate in the liquor would have an adverse effect on the absorption, and this is confirmed.

TABLE I.—*Experimental Data Obtained on 1 in. diam. Falling Film Column*

Liquor composition	Absorption coefficient (lb. mol./hr. sq. ft. atm.)
0·047 M NaOH	0·45
0·056 M Na_2CO_3	0·40
0·056 M Na_2CO_3 + 0·088 M $NaHCO_3$	0·36
0·056 M Na_2CO_3 + 0·5 M $Na_2S_2O_3$	0·35
0·056 M Na_2CO_3 + 0·3 M Na_2SO_4	0·37
0·047 M Na_2CO_3	0·38
0·047 M Na_2CO_3 + 2·9% Fe $(OH)_3$	0·38

In the "regeneration" stage, the main reactions are probably :—

$$2Fe_2O_3 + 6NaHS \longrightarrow 2Fe_2S_3 + 6NaOH$$
$$2Fe_2S_3 + 3O_2 \longrightarrow 2Fe_2O_3 + 6S$$

Reactions other than those above, and complex in nature, also occur.

Much of the early work was confused by changes in the activity of the catalyst and by incomplete knowledge of the effect of these other reactions on the process as a whole. Catalyst activity varies considerably, but it was not initially appreciated that the catalyst plays only a small part inside the actual absorption chamber. The results in Table I demonstrate this, the coefficient being unaffected by the presence of catalyst because of the low contact time in the absorption chamber. Substantially all the oxidation therefore takes place in the regeneration tank. If the tank were large enough, a less active catalyst could be used without significantly affecting the absorption.

Various side-reactions were studied and also the oxidising action of the air which is bubbled through the liquor. If insufficient air is supplied the liquor becomes reduced and absorption is impaired ; its colour changes from chocolate-brown to green. If excessive oxidation occurs then side reactions[1] increase and alkali usage rises.

Process requirements

Briefly, the laboratory work pointed to the following requirements, some of them conflicting :

(*a*) To obtain good absorption in the scrubbing chamber :

  (1) Large liquor surface area per unit volume.
  (2) High alkalinity (preferably caustic soda).
  (3) Low content of dissolved salts in the liquor.

(*b*) To obtain economic regeneration in the tank :

  (1) Low alkalinity.
  (2) Low degree of aeration.
  (3) High catalyst content.
  (4) High oxygen transfer rate.

Requirements (*b*1) and (*b*2) are to minimise the formation of products other than sulphur. Requirements (*b*3) and (*b*4) are to minimise the size of tank.

Pilot Plant Work

The pilot plant was built around a 2 ft. square scrubbing tower. The liquor was circulated through the tower from a storage tank. Smaller tanks were installed for catalyst preparation.

The tower was initially 15 ft. 6 in. tall, and contained 10 ft. of glass plate packing, the plates set at 45° from the vertical. The top section contained a liquor distributor and spray eliminators.

The sulphur formed in the process collected as a scum on the surface of the liquor in the tank and was removed at intervals.

Work on this pilot plant showed that it would not reduce the concentration of hydrogen sulphide from 300–400 p.p.m. at inlet down to less than 100 p.p.m. at air rate of 700 cu. ft./min. It was then decided to remove the packing and to use the tower as a spray chamber. Under these conditions the hydrogen sulphide removal was similar to that obtained when the packing was present, whilst the pressure drop imposed on the air was lower. As a result of these experiments, and of other considerations, it was decided to develop spray chambers rather than packed towers. An important factor in these considerations was the reduced resistance to the air-stream.

At this stage of the work the results were difficult to reproduce and the effect of some aspects of the liquor analysis was not understood. Only first order effects could therefore be relied upon.

Fig. 1 illustrates the pilot-plant in its final form. The 2 ft. square tower had an effective height of 12 ft. and had seven full-cone spray jets spraying down the central axis. The pump was capable of delivery at 100 lb./sq. in., to permit a wide range of spraying pressures. The liquor flow from the tower was measured over a weir.

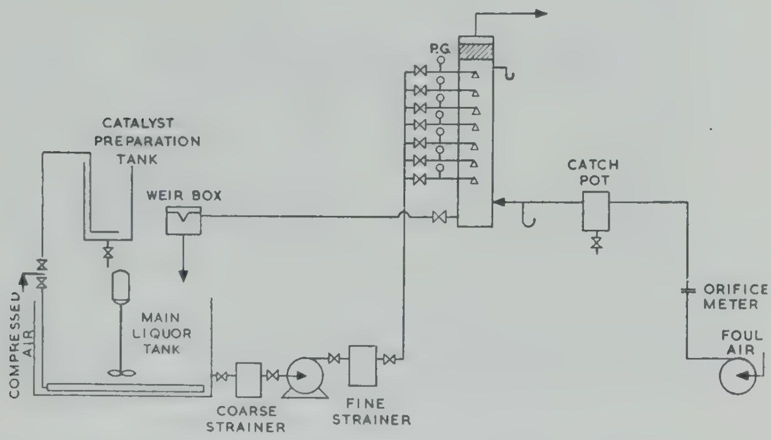

Fig. 1.—*Pilot-plant for hydrogen sulphide scrubbing*

Sparge pipes used for aerating the liquor in the tank were replaced by horizontal canvas sleeves in the bottom of the tank. Because of the finer bubbles, these sleeves gave improved aeration with less air and with only a small increase in the air pressure required.

Variables considered

It was found that increasing the air velocity up the tower improved the performance coefficient, presumably by reducing the falling velocity of the droplets thereby increasing the hold-up of droplets in the chamber, and also by increasing the average relative velocity between the droplets and the air.

The effect of liquor alkalinity was studied and it initially appeared that this had a first order effect, since graphs of absorption coefficient against time and alkalinity against time were very similar. It was later discovered, however, that this was because at times when the alkalinity to phenol phthalein was increasing (due to excess soda corrections) a good performance was obtained, whereas a falling alkalinity tended to give a poor performance. These effects were due to changes in the ratio of sodium carbonate to sodium bicarbonate, under non-equlibrium conditions. When the alkalinity was permanently raised the improvement was less marked.

Varying the catalyst concentration showed that if it fell below about 1%, reduction of the catalyst led to a considerable drop in performance. The concentration above 1% had only a marginal effect, confirming the fact already noted that if the catalyst concentration is sufficient to oxidise the liquor completely in the tank before it returns to the scrubbing chamber there is no advantage in raising it.

By varying the volume of liquor in the tank, it was shown that tank size and catalyst concentration are interdependent and have to be fixed to give a negligible sodium hydrosulphide concentration at the sprays.

Variation of the air flow to the aerator sleeves was complicated because as the air quantity was reduced the catalyst tended to settle out of suspension. A stirrer was therefore installed and it was then found that the air flow could be considerably reduced without affecting the performance.

The effect of varying the spraying pressure was investigated, the total liquor flow being kept approximately constant by reducing the number of jets operating as the pressure was increased. An improved performance was obtained, presumably because of the smaller droplet size at the higher pressures, but it was found that with the type of jet used, increasing the pressure from 20 lb./sq. in. to 88 lb./sq. in. (*i.e.* by a factor of 4·4) improved the performance coefficient by a factor of only 1·4. Because, when other factors are constant, the coefficient is very approximately proportional to the total liquor flow, it follows that if the pressure is increased while the flow is reduced, to maintain a constant pumping horsepower, a poorer performance will result. Subsequent spraying pressures have therefore been kept fairly low. The pressure-flow characteristics of various types of jets were examined, with a view to obtaining jets which would give a fine spray whilst not tending to block.

Factors associated with the position of the jets in the tower had to be considered. Because the quantity of liquor to be sprayed would be large, necessitating a large number of jets, and since the sprayed droplets were known to be most effective within a few feet of the jets, it was decided at that time that full-scale plants should utilise scrubbing towers in which the jets would be evenly spaced throughout the volume.

At this stage it was considered that sufficient data were available to design a prototype full-scale unit.

Scale-Up Considerations

It was clearly difficult to design a spray chamber from wetted-wall column experiments since, apart from other considerations, it is almost impossible to predict the surface area of the droplets in a spray chamber. The laboratory wetted-wall column results were therefore useful only to compare the absorption characteristics of liquors of differing analyses and to elucidate the fundamentals of the process.

It should, however, be possible to design a full-scale spray chamber from results obtained in a pilot spray chamber. This was effected in the present project with some success although the first (vertical) full-scale unit fell a little below design specification.

As has already been stated, it was decided that the first full-scale unit would be similar to the pilot unit, *i.e.* a vertical tower. Having fixed the duty, the factors to be fixed in the design were :

(1) the volume of the chamber,
(2) the shape of the chamber,
(3) the liquor circulation rate and spraying pressure,
(4) the geometrical positioning of the jets,
(5) the type and size of jets, and
(6) the size and shape of the regeneration tank.

Considerable thought was given to factors (4) and (5) but these will not be considered here except to note the decision to space the jets uniformly throughout the volume and to use jets similar to, but larger than, those used on the pilot-scale.

The volume of the chamber was considered by means of a volumetric absorption coefficient defined as follows :

$$K_1 = \frac{\text{lb. mol. } H_2S \text{ removed}}{(\text{hr. cu. ft.})\ (\text{log mean concentration } H_2S \text{ in atmospheres})}$$

This coefficient has been frequently used in this project and has been the main measure of plant performance. It is clear, however, that it will vary with the type of jet, the pressure of spraying (*i.e.* the droplet size), the shape of tower, and particularly with changes in the liquor circulation rate per unit volume.

Another coefficient can be defined in terms of liquor circulation, and has also been used in this work, in combination with K_1 :

$$K_2 = \frac{\text{lb. mol. } H_2S \text{ removed}}{(\text{gal. liquor})\ (\text{log mean concentration } H_2S \text{ in atmospheres})}$$

It should be noted that K_2 equals K_1 divided by the liquor flow per hour per cu. ft. of chamber and is probably a more significant coefficient than K_1.

K_2 also varies with the type of jet, the pressure of spraying (*i.e.* the droplet size), and also with tower volume and shape.

It should be noted that the use of the log mean H_2S concentration as the " driving force " in both K_1 and K_2 implies two assumptions :

(1) that countercurrent or " cross-flow " conditions occur, *i.e.* that the air passes as a plug through the chamber, without " back-mixing ", and

(2) that the liquor has no appreciable equilibrium vapour pressure of hydrogen sulphide.

It is clear that neither of these assumptions is really justified. Both assumptions will, however, be more valid for the pilot unit since :

(*a*) the shape ratio of height to width was much greater in the pilot unit (6 : 1) than in the large vertical units, and

(*b*) because in the pilot unit the outlet concentration of hydrogen sulphide was always quite high, any vapour pressure of hydrogen sulphide exerted by the liquor would have little effect.

On these grounds one would expect the pilot unit to operate more " efficiently " than the large units. A disadvantage of the pilot unit was, however, that there was a very large wall effect which must have eliminated the spray droplets before they travelled very far. The wetted walls would themselves absorb hydrogen sulphide but probably not very effectively.

In a large vertical tower of " squat " dimensions of the type built and described below, the shape ratio was low and it was realised that there might be a very small concentration gradient through the tower. In the most adverse circumstances the air in the tower might be mixed from top to bottom, particularly considering the eddy currents imposed by the sprays. The average concentration of hydrogen sulphide would then be equal to the outlet concentration and the driving force would therefore be much lower than the log mean of the inlet and outlet concentrations. Although this was not proved, it seems likely that something approaching this condition occurred in the full-scale vertical tower (see below).

The coalescence of droplets was also considered, particularly since the concentration of falling droplets at the bottom of a full-scale tower must be extremely high, and would be higher if a tall thin tower were used instead of a squat one. Ultimately, if a very tall tower were required (for shape ratio reasons) it might have to be split into two or three towers in series in order to reduce the concentration of droplets at the bottom. Such an arrangement would be complicated for the volume of air to be handled in this project, and the resistance to air flow would probably be high. The towers had therefore to be reasonably squat.

Prototype Vertical Full-scale Unit

The prototype vertical full-scale unit was basically similar to the pilot plant and a description has already been published.[1]

The volume of the absorption tower was calculated from the volumetric coefficient K_1 obtained on the pilot plant, the design throughput being 40–45 000 cu. ft./min. This meant that a group of six such towers would be required for a single factory unit.

The calculated volume of 6144 cu. ft. was obtained by making the tower 16 ft. square, with an effective scrubbing height of 24 ft. The liquor flow per unit volume was decided by consideration of the values of coefficients K_1 and K_2 obtained on the pilot plant (and was actually lower than the typical pilot plant figure). It was not practicable to use the same size of jet as on the pilot plant, but a larger size of the same type was used. The jets were arranged in 12 horizontal layers uniformly spaced throughout the tower.

The dimensions of the liquor regeneration tank were calculated by scaling-up the minimum volume (from pilot plant experience) on the basis of plant throughput. The depth of liquor was made as great as possible, since it was thought that this would facilitate aeration. The total surface area of aerator sleeves was scaled-up proportionally to the throughput. The same type of sleeve aerator was utilised on this plant as on the pilot plant, but the sleeves were made as large as practicable.

The performance of the unit (as measured by the volumetric coefficient K_1) was found to be comparable to that obtained on the pilot plant (see Table II below) and was considered reasonably satisfactory. The value of K_2 was also reasonable. The concentration of hydrogen sulphide was reduced from 300 p.p.m. to 20–30 p.p.m. (It had been hoped that the outlet concentration would be 10–20 p.p.m.)

TABLE II.—*Comparison of Results Obtained on Pilot Plant and Prototype Units*

Plant	Gals per hour liquor	Flow (g.p.h.) per unit volume	Liquor pressure (lb./sq. in.g.)	K_1	K_2	Remarks
Pilot plant	2 100	42	20	3·5	0·084	7 jets.
Pilot plant	2 160	43	88	5·0	0·12	4 jets.
Pilot plant	2 400	48	40	4·4	0·091	7 jets.
Vertical full-scale . . .	180 000	29	15–30	3·0	0·10	Typical result.
Vertical full-scale . . .	189 000	31	20–30	4·0	0·13	Exceptionally good result.
Horizontal full-scale . .	480 000	25	31	3·1	0·12	Without grids.
Horizontal full-scale	480 000	25	31	3·2	0·13	With grids.
Horizontal full-scale one-third of chamber only	213 000	33	39·5	5·0	0·15	K_1 based on one-third volume.

The liquor in this unit contained a different ratio of carbonate to bicarbonate compared with that in the pilot plant. This had an influence on performance. A satisfactory reason for this difference has not been found, particularly in view of the fact that the liquor analysis in the horizontal unit (see below) is similar to that in the pilot unit.

It was difficult to obtain a reliable sample of the treated air because liquor entered the sample lines and absorbed some of the hydrogen sulphide in the sample. Air leaks were also troublesome. This was a feature of the work, and indicated for some time that the performance of the unit appeared to be better than it actually was.

It was also very difficult to take samples of air at different levels in the tower to check whether there was a gradient of hydrogen sulphide concentration through the tower, and to estimate the degree of vertical mixing of the air that was occurring. This work confirmed the view that considerable mixing was occurring, but the results were to some extent suspect because of liquor entering the sample lines in spite of very elaborate precautions to prevent this.

Consideration of a Horizontal Scrubbing Unit

An estimate of the cost of operating this unit showed that the biggest single item was the cost of liquor circulation. Thought was therefore given to reducing this cost, and the horizontal scrubbing unit described below was conceived. The basic idea was to save the power required to lift the liquor to the sprays in the vertical tower by spraying upwards from ground level at an angle of about 45°. The air in such a unit would move horizontally, the liquor movement being largely horizontal, at right angles to the air stream. The cross-section of the horizontal chamber would be " fitted " to the spray profile resulting from jets sited to spray across the chamber from opposite sides. It was decided to use orifice-type spray jets, which give a flat fan-like spray. Tests were carried out on three sizes of orifice-type jets and photographs were taken of the spray profile from one of them. From these photographs the optimum cross-section for the chamber was decided upon. The length of the chamber could then be fixed by assuming a volumetric coefficient K_1 (equal to that obtained in the vertical unit) and the liquor flow from the corresponding value of K_2. The chamber would have the general appearance of a Nissen hut.

The horizontal unit appeared to have several advantages :

(1) it should save about 40% of the power required for liquor circulation through vertical towers.

(2) One horizontal unit could be equivalent to three or more vertical units and should cost much less, since it could be of light construction.

(3) Orifice jets could be used, which would not easily block and because of their position they could be easily removed from the chamber.

(4) The difficulties of air-liquor separation which had occurred at the bottom of the vertical tower would not arise.

(5) The horizontal chamber could easily be baffled to restrict back-mixing.

A disadvantage was that it would be difficult to obtain a reliable indication of the performance of a full-scale unit from small-scale experiments. It was therefore decided to take the risk of building a unit equivalent in throughput to three of the vertical towers, without further small-scale experiments.

Prototype Horizontal Full-scale Unit

The prototype horizontal unit was designed for a gas flow of 125 000 cu. ft./min., *i.e.* three times that of the vertical tower.

The chamber is 55 ft. long, 30 ft. wide, and 15 ft. high, the cross-section being approximately semicircular. A general photograph of the installation is shown in Fig. 2. The gas velocity through the chamber is 5·9 ft./sec. After the unit had operated for a short time, two grids were installed across it dividing the chamber into three equal sections in order to promote stage-wise absorption. These grids consist of lengths of timber, attached vertically to a framework, the area of timber being half of the cross-sectional area of the chamber. It was considered that the gas velocity of 11·8 ft./sec. through these grids should suffice to prevent back-mixing of the air from one section of the chamber to the other.

The liquor is supplied by a row of 140 jets along each side of the chamber at ground level. The jets are slightly inclined to throw the spray against the direction of flow of the air. The jet fittings are designed so that the jets may be removed from outside the chamber. The liquor pressure at the jets under normal operating conditions is about 30 lb./sq. in. g. and the total liquor flow 8000 gal./min. By bringing a standby pump into use, the liquor pressure at the jets may be increased to 39 lb./sq. in. g. and the flow to 9200 gal./min.

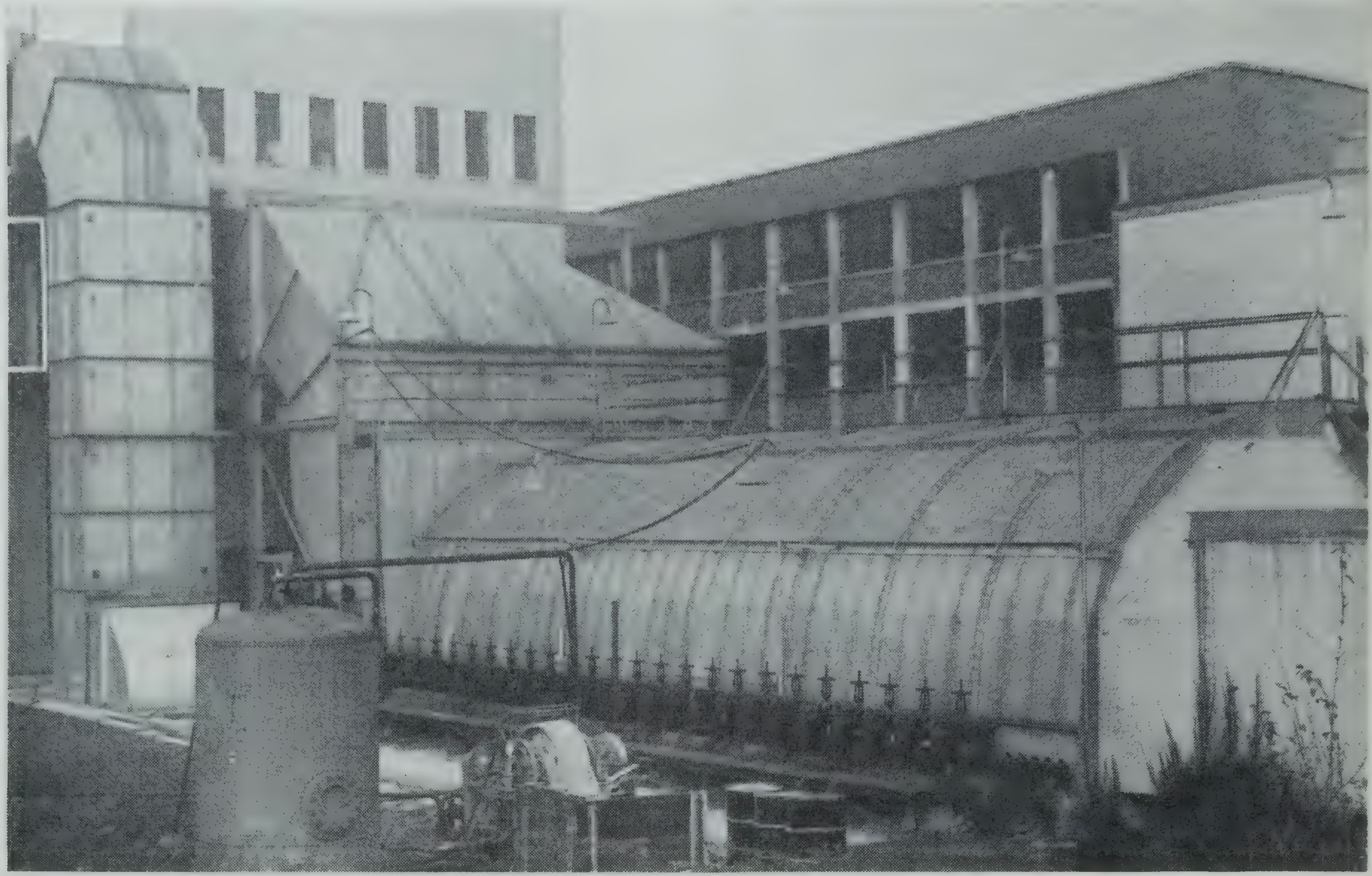

Fig. 2.—*Horizontal hydrogen sulphide scrubber*

The return liquor flows into a large circulating tank, with the liquor surface approximately at ground level. Aeration and stirring of the liquor is by nylon-sleeve aerators.

The sprayed gas passes through spray eliminators consisting of vertical stainless steel plates at approximately 1 in spacing, each plate having six changes of direction. The liquor carry-over through the eliminators is negligible.

Results

The plant has been operating for eight months and the results obtained are as follows.

Under normal liquor flow conditions, before the grids were installed, the hydrogen sulphide in the air was reduced from 300 p.p.m. to 17 p.p.m. on average giving a value of $K_1 = 3{\cdot}1$ lb. mol./hr. cu. ft. atm. After the introduction of the grids the outlet concentration of hydrogen sulphide was 13 p.p.m., giving a coefficient of 3·2 lb. mol./hr. cu. ft. atm. During short periods when the liquor flow was increased to 9200 gal./min., the outlet concentration of hydrogen sulphide was reduced to 5–10 p.p.m., giving a value of $K_1 = 4{\cdot}2$ lb. mol./hr. cu. ft. atm.

Approximately 0·4 mol caustic soda have been used per mol. of hydrogen sulphide removed. This is greater than in the vertical tower but it is not yet clear why this is so.

The pressure-drop over the length of the chamber, excluding the eliminator plates, is 0·3 in. H_2O and the introduction of the grids caused no significant increase in this. Measurements of the static pressure in the chamber with the liquor circulating but no gas flow through it indicated that a considerable proportion of this pressure drop is due to the effect of the sprays being directed into the airstream, and could be influenced by changing this feature. The pressure drop across the spray eliminators is normally 0·2 in. H_2O but fouling of the plates has on occasions caused this to rise.

Typical results obtained at the three main stages of the project are summarised in Table II.

Discussion

The consistency of the values of K_1 and K_2 (as indicated in Table II) despite large scale-up factors (up to 390 based on chamber volume), is notable in view of the fact that the full-scale horizontal unit is significantly different from the pilot and full-scale vertical units. The horizontal unit is of a different shape, the direction of movement of the droplets relative to the air stream is different, and a different type of jet is used.

When the decision was made to construct the horizontal unit these differences were appreciated but it was considered that coefficients K_1 and K_2 which would be obtained, should be approximately equal to those on the small scale if the proposed jets gave a droplet-size similar to the previous ones used, and if the liquor flow per unit volume was maintained approximately as in the vertical unit.

The first condition was checked merely by visual observation when choosing the jets, and there was, of course, a possibility of a different degree of coalescence in one plant compared with another. Another factor that was difficult to allow for was the knowledge that the activity of the droplets as absorbers almost certainly falls off rapidly as the distance from the jet increases, and this effect might have more influence on the performance of the horizontal chamber than on that of the vertical tower. It was considered, however, that the above differences would not amount to a great deal. These forecasts have in fact been justified by the results obtained.

Finally, it should be stated that in taking a technical risk in building the first horizontal unit it was known that if the performance had not reached the specified level, this might have been corrected by increasing the liquor circulation rate, *i.e.* by installing extra pumps. A possible safety factor such as this allows a difficult scale-up operation to be approached with more confidence.

Acknowledgments

The authors would like to express their thanks to the Directors of Courtaulds Ltd. for permission to publish this paper. They also wish to acknowledge the contribution made to this project by some of their colleagues and the factory staffs.

Symbols Used

K_1 = overall mass transfer coefficient, volumetric basis,

$$\left(\frac{\text{lb. mol.}}{\text{hr. cu. ft. atm.}}\right)$$

K_2 = overall mass transfer coefficient, liquor flow basis,

$$\left(\frac{\text{lb. mol.}}{\text{gal. atm.}}\right)$$

Reference

[1] Pratt, D. C. F., and Rutherford, A. *Chem. & Ind.* (*Rev.*), 1955, p. 1281.

The manuscript of this paper was received on 30 November, 1956.

THE EFFECT OF SCALE ON AIR-SEPARATION PLANT DESIGN

By P. M. SCHUFTAN, Dr.Phil., M.I.Chem.E.* and J. FORREST, B.Sc., A.M.I.Mech.E.*

SUMMARY

The effect of the size on the technical and engineering design of air separation plants is discussed, based on practical experience and theoretical considerations.

The performance and type of compression and cold-producing machines and of heat and mass-transfer equipment is determined by the scale of the plant, and an operating cycle has to be so chosen as to enable the use of efficient and economic plant components which can be made available.

Introduction

The separation of air by liquefaction and rectification is a purely physical process and essentially involves heat- and mass-transfer operations. Heat exchangers are necessary for the recovery of the cold in the separated products—oxygen and nitrogen—and for heat transfer to the air to be separated, while the rectification is a mass-transfer process. In addition, refrigeration has to be supplied to compensate for unavoidable losses in the heat exchangers and for heat influx through the insulation. Extra cold is required when liquid products have to be obtained. Generally the air to be separated is also used to provide the refrigeration.

The necessary corollary to the steps of heat- and mass-transfer and of cold production is the compression of the air in reciprocating or rotating compressors to pressures which—according to the operating cycle used—lie between 4·8 and 240 kg./sq. cm.

In many types of air separation plants equipment has to be provided for the removal of carbon dioxide and moisture from the air in order to avoid obstructions during the cooling process.

Type of Régime

Johnstone and Thring have defined three types of régime : " dynamic ", " thermal " and " chemical ", as the determining factors of chemical engineering processes.

The separation of air takes place essentially under a dynamic régime if one ignores the effect of the temperature differences as a driving factor in the heat- and mass-transfer operations. The determining factors for scaling-up or down are therefore essentially the Reynolds and Froude numbers.

For a heat exchanger, the heat to be transferred and the permissible temperature difference determine the product $F \times k$ where F is the heat-transfer area and k the overall heat-transfer coefficient. k is a direct function of the Reynolds number and by trial and error a hydraulic diameter and length of tube are found which satisfy both the permissible pressure-drop and the constructional requirements.

For the same basic conditions, but working on a different scale all that is necessary, in principle, is to scale the number of heat-transfer passes. Should a limitation be imposed by constructional difficulties F or k can be altered as long as the product $F \times k$ is constant. For instance, tubes with extended surface may be used or a non-tubular type of exchanger specified. An increase of k will only be possible in exceptional cases without exceeding the permissible pressure-drop.

Design problems resulting from the scale of air-separation plants are therefore to be expected to arise in the machines for compression and cold production, the purification equipment (if any), and the heat- and mass-transfer system. Accessories, such as electric motors, water pumps, etc. are neither peculiar to the processes, nor do they present any particular problem in scaling-up.

Range of Units

The scale range of industrial air-separation plants is rather wide with power consumptions between 20 and 5000 kw. per unit. Within these limits, the specific cold loss per volume of air processed varies in the ratio of about 5 : 1 and the available means of producing the required amount of cold determines the operating cycle which can be used at any particular scale. This means that we are not normally concerned with scaling-up a particular operating cycle over a very wide range but rather the operation of scaling takes place within certain distinct levels. The limits of these are essentially determined by mechanical limitations and economic considerations and less by actual difficulties in chemical engineering design. The technical design on each level further depends on whether the plant has to produce gases only, or whether having regard to the particular location of the plant, storage and transport of liquid products is more economical.

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* British Oxygen Engineering Limited, Edmonton, London.

In small plants the cold is produced by isenthalpic expansion (nozzle expansion), which while thermodynamically inefficient, can be carried out without losses and without elaborate devices. The opposite applies to the isentropic expansion in reciprocating or rotating expansion machines which must be used in larger plants and is generally necessary for liquid production.

The thermodynamic efficiency of expansion machines rises from 50–60% for the smallest size rated at about 3 kw. to over 80% for machines of 100 kw. or more.

Simplifying conditions greatly, we therefore obtain roughly the scheme outlined in Table I.

TABLE I.—*Effect of Operating Level on Technical Design*

Plant size	Products	Operating pressure (atm.)	Cold production
Up to 50 kw.	Gases	~ 60	Isenthalpic.
Over 50 kw.	Gases	4·8–17	Isentropic (turbines).
Over 50 kw.	Liquid	140–180	Isentropic (reciprocating expansion engines)

In each of these cases the technical design of the plant components is very different; thus a simple scaling-up is not possible.

Economic Aspects

Operating costs of small plants are essentially controlled by labour charges[1] while in medium-sized plants capital charges and in large plants power costs are dominating. Small plants thus have to be simple and possibly automatically controlled but need not necessarily be thermodynamically efficient.

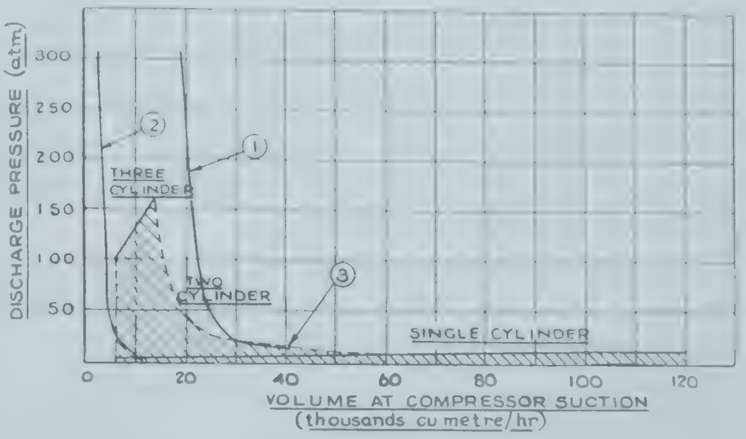
In larger plants, considerable savings are possible by ensuring long, continuous operating periods, thus reducing or eliminating the need for standby units. For the largest plants, a cycle of very high thermodynamic efficiency is a prime condition.

Effect of Scale on Design

In the following the effect of scale on the technical design of the particular plant components is considered in more detail:

Compressors

Fig. 1 shows potential pressure-volume ranges for reciprocating and centrifugal air compressors which are the two types most suitable for the particular cycles.



Curve 1.—Reciprocator
Curve 2.—Reciprocator boosted at 6 atm.
Curve 3.—Radial turbo-compressor

Fig. 1.—*Capacities of rotary and reciprocating compressors*

In general, reciprocating compressors are limited to about 1000 b.h.p. per crank because above that size the motion work becomes too massive, and with more than 6 cranks, the machine itself would become too large. Therefore the maximum practicable size of a unit is about 6000 b.h.p. In addition, as the first-stage cylinder increases in size, the distance between cranks imposed by the cylinder diameter becomes too great even when this effect is partially offset by the use of horizontal opposed cylinders.

Curve 1 of Fig. 1 shows the approximate trend which such a large reciprocating compressor might take, ranging from a low pressure, 6-crank, single-stage unit, aspirating 73 000 cu. metres/hr. to a 6-crank, 6-stage unit aspirating 20 000 cu. metres/hr. and delivering at, say, 300 atm. Any compressor on this curve would probably require some sort of unwelcome mechanical compromise apart from the fact that the size and cost of the machine with its associated foundations and buildings would be excessive.

Therefore another solution is sometimes used, namely, the supercharging of the first-stage cylinder. For instance, if a compressor on curve 1 is functionally split into a turbo-compressor aspiring the same volumes and delivering at 6 atm. to the first-stage cylinder of a reciprocating compressor, the latter unit only requires to aspirate the volumes shown in curve 2 in order to be equivalent in discharge capacity to a unit on curve 1. Thus the heavy and expensive large bore cylinders can be eliminated, and the designer can reduce the stroke, increase the speed, or shorten the distance between cranks.

The overall capital cost of such an arrangement is substantially reduced. However, the lower efficiency of the turbo compressor reduces the efficiency of the entire set by about 2%. It is usually worthwhile to reduce the capital cost at the expense of running cost. Many combinations are possible : in general, the cheapest capital cost results from the maximum supercharge, and the smallest reduction in efficiency results from the minimum super-charge.

Actual units use a single-wheel blower delivering air at 1·4 atm. abs. to a 5-stage compressor. The suction volume is 9000 cu. metre/hr. and delivery pressure is 200 atm. A second design uses a multi-stage rotary compressor delivering air at 6 atm. to a 4-stage air compressor. The suction volume is 18 000 cu. metre/hr. and the delivery pressure 200 atm.

Such compressor combinations cover requirements for liquid oxygen plants up to 50 or 100 tons/day but still larger plants would tend to be gas plants with low pressure compression units. Curve 3 shows the range which would be possible for radial turbo compressors.

The " multi-cylinder " high pressure units are only shown as a matter of interest. Their efficiency is too low to have a practical application for air-separation units. In actual practice, a delivery pressure of 5 atm. may be adequate, and the output range up to 120 000 cu. metre/hr. covers all immediate needs.

Axial compressors have not such suitable characteristics as radial units for air separation processes, but they could be used, if larger plants were contemplated, for a range from, say, 100 000 cu. metre/hr. upwards.

Altogether scaling-up turbo-compressors presents no fundamental problems, and no special developments have been envisaged to permit further scaling-up beyond the range shown. Increased throughout is achieved simply by increase in size limited by physical considerations or by increase in number of units.

Expansion machines

With regard to air-expansion machines, a similar process takes place, whereby as the plant size increases a change is made from reciprocating machines to centrifugal machines, coinciding also with the change from the high-pressure liquid-product plant to a low-pressure gaseous-product plant.

The reciprocating engine may be limited in cylinder size by the problem of low temperature cylinder lubrication. No difficulty has been found with engines so far built, because the heat produced by piston-ring friction appears to balance the cooling effect of the expanding air, even although this may be at −160° C. In consequence, an ordinary light-grade mineral oil will spread uniformly over the cylinder wall. There is some evidence that with large cylinders this condition may no longer hold, but, in any event, valve gear would tend to present complications for large bore cylinders. Two-stage expansion presents the same lubrication problem in the colder low pressure stage, so that scaling-up is best achieved by increase in speed or in number of cylinders in parallel. Mechanically a reciprocating engine could be designed to develop up to 200 h.p. per cylinder, so that, as a 4-cylinder unit running at, say, 240 r.p.m. it could expand about 25 000 cu. metre /hr. from 200 atm. to 5 atm. in a single stage which is much more than at present required.

However, with the change to a low pressure process for a large gas plant, the use of a reciprocator becomes out of the question because of the size of cylinders required, but a rotary machine can now be used.

Air expansion turbines may be of the axial or radial types, and both are used. Designs cover a very wide range of speeds, throughputs, and pressures. There are no particular problems associated with scaling, although there are many special design features otherwise.

It will be seen that for larger plants, mechanical design has followed normal practice of operating at faster speeds in order to reduce the proportionate capital cost, and no special difficulties have been involved for the capacities envisaged. The interesting feature lies in the fact that changes in plant duty and increased scale have compelled the development of processes which can utilise rotary machines with their manifold economic advantages.

Coolers

In low pressure processes, the cooling of the compressed air is more economical by direct contact with water in scrubbers than indirectly in tubular coolers. Here, scaling-up involves the well-known problem of water distribution for increasing scrubber sizes.

Heat exchangers

The scaling up of heat exchangers of the tubular, non-tubular, or regenerative type does not usually present any special difficulty, except in very large units where it may be necessary to provide several units in parallel. Special care is then required to ensure uniform loading of each unit.

Carbon dioxide elimination

In small plants, counter-current washing of the air with caustic soda solution between compressor stages is commonly used, with provision of a large storage vessel at the base of the scrubber and intermittent renewal of the spent lye. In large plants such an arrangement is expensive. The cumbersome batch operation is then replaced by continuous lye-injection and withdrawal, eliminating the large storage pressure vessel altogether. The size of the scrubber itself is reduced by operating in co-current instead of counter-current flow which is possible as there are no limitations imposed by chemical or physical equilibrium conditions.

Driers

Separate driers are required in any plants in which the use of regenerators or reversing exchangers is not possible. Moisture removal takes place at the operating pressure in a vessel filled with activated alumina and a second identical vessel for alternate use is provided, which is regenerated by passing warm and dry nitrogen through it in an upward direction. For large sizes there is a mechanical limitation with regard to the practicable diameter of these vessels, which must be large enough to prevent up-lifting of the adsorbent bed during reactivation. In small driers all the heat required for reactivation is provided as sensible heat of the reactivating gas, and as its maximum temperature is limited the flow rate becomes rather high.

In large driers it is feasible to supply a considerable fraction of the heat directly to the wall of the vessel, either by an electric heater or an oil jacket. With such shell heaters the flow rate of the reactivating gas can be much reduced and the vessel diameter can be kept relatively small without exceeding the lifting velocity, and in this way a mechanical limitation is overcome which would otherwise arise when scaling-up driers of relatively small diameter.

Efficiency of purification

As already mentioned, long operating cycles must be achieved in medium and large plants and a simple scaling-up of the equipment for carbon dioxide elimination and drying would therefore normally not be adequate. Additional scrubbing surface has to be allowed for the carbon dioxide elimination. The efficiency of the driers can be increased by operating them at reduced temperatures, thus reducing their load while keeping their size normal.

When long operating cycles are necessary, meticulous provisions have to be made for the efficient elimination of other air contaminants, especially hydrocarbons, as their accumulation in the air separation unit can constitute a danger.

Rectification columns

In small plants, packed columns are quite suitable and H.E.T.P's of less than 3 in. are industrially achievable. In view of distribution difficulties, tray columns must be used in medium and large size plants. When scaling-up tray columns the vapour load is roughly proportional to the square of the diameter, while the weir loads become relatively larger. This would normally require an increase in the tray spacing but this is only possible to a limited extent as cold losses have to be kept at a minimum. In large plants, the hydraulics of the column therefore require special attention and suitable provisions have to be made to reduce the hydrostatic liquid heads so as to keep the column diameter within the limits imposed by the vapour velocity.

Conclusions

The air separation process takes place essentially under a dynamic régime. The scale range is between 20 and 5000 kw./unit, but the operating cycle—and therefore scaling-up problems—depends on and vary with particular size ranges. These are determined by the cold requirements of the plant and by available means of covering these cold requirements. Turbo-machines are used for compression and cold production where possible.

Scaling-up problems are mainly mechanical or due to economic considerations while there are no special chemical engineering difficulties.

Reference

[1] Schuftan, P. M. *Proc. 8th Int. Congr. Refrig.*, p. 136.

The manuscript of this paper was received on 3 *December*, 1956.

DISCUSSION OF PAPERS PRESENTED AT THE THIRD SESSION

Mr. S. P. S. ANDREW mentioned absorption with chemical reaction where the absorption mechanism was affected both by diffusional transfer in the liquid phase and by fairly rapid chemical reaction. Examples were the absorption of carbon dioxide into caustic liquor and into ammonia liquor. If the concentrations of the liquor and gas were in the right range, and if the liquor turbulence was sufficient, then the absorption rate per unit area of liquid/gas interface was independent of the turbulence. The liquid film transfer coefficient was also independent of the turbulent conditions, provided turbulence was adequate, so that if the liquid/gas interfacial area was known, scale-up over a very large range, say, a range of 10 000/1 in surface area, could be effected with some confidence.

Knowledge of the effective interfacial area was not easy to acquire for random-packed columns, but for columns which were packed with, say, stacked rings or grids, with an adequate liquid flow and good distribution, the interfacial area was reasonably well known, so that scaling-up a ring-packed tower used for absorbing carbon dioxide in caustic solution or in ammonia liquor was possible over a very wide range. Experiments could be made with apparatus having only 100 sq. cm. of interfacial area and the results scaled-up to plant having 1 000 000 sq. cm. of interfacial area with a fair degree of confidence to within a factor of 1·5.

His second point concerned equation (3) (p. S76) of Mr. Hoftyzer's paper. As the author had pointed out, c appeared to vary as the packing size was changed. It would appear that c was proportional to (packing size)$^{0\cdot 8}$. If $D =$ characteristic packing dimension, $c \propto D^{0\cdot 8}$.

It occurred to him that it was possible that the characteristic linear dimension on the left-hand side of equation (3) had not been chosen appropriately. If the fully wetted case were taken, where $a = a_p$, this linear dimension reduced to $1/a_p$. He suggested that this characteristic dimension should be taken as proportional to the thickness of the liquid layer on the packing, because it was the thickness of this layer which largely determined the turbulence. He therefore suggested that the characteristic linear dimension in the matter-transfer Nusselt number should be taken as

$$\left(\frac{u\mu}{a_p\,\rho g}\right)^{\frac{1}{3}}$$

This quantity had the dimension of length and was proportional to the thickness of the liquid layer. The modified correlation would now be :

$$\frac{k_l}{D_l}\left(\frac{u\mu}{a_p\,\rho g}\right)^{\frac{1}{3}} = c^*\left(\frac{u\rho}{a_p\,\mu}\right)^{1.1}\left(\frac{\mu}{\rho\,D_l}\right)^{0.4}$$

The index of the Reynolds number had been altered to maintain $k_l \propto u^{0\cdot 8}$. The characteristic linear dimension in the Reynolds number did not need any alteration, because the existing Reynolds number was, in effect, based on the average mass velocity in the liquid layer multiplied by the layer thickness. It would be found that c^* was proportional to $D^{-0\cdot 2}$, for the calculation of c.

Although the effects of maldistribution could not be calculated precisely, an estimate of their importance could be made. With a large number of transfer units in a tower, accompanied by a large change in liquid composition and liquid back pressure, slight maldistribution would have a pronounced effect on the efficiency of the tower. A numerical check could be made to see whether or not maldistribution was likely to be important.

Mr. MAX LEVA said that the deviations which were shown in Fig. 1 of Mr. Hoftyzer's paper were plotted logarithmically, and whenever one saw such a plot one suspected that some factors had not been accounted for. He thought that the correlation in equation (3) on the same page was not quite adequate.

One omission was the effect of the ratio of the height of the packing to the column diameter. The author measured the thickness of the liquid streams along the packing by means of conductivity measurements. This liquid thickness would be expected to increase as the column height increased, or in other words as the contact time of the liquid down the column increased. This would not normally show in ordinary liquid distribution studies, but the overall surface area of packing which was wetted would be reduced when the streams coalesced.

When carbon dioxide was absorbed by sodium hydroxide liquor there was no back pressure of the carbon dioxide over the solution and the driving force was the partial pressure of the carbon dioxide only. It was easily shown by experiments that the values of $K_G a$ were independent of the gas rate when operating below loading of the packing, and depended only on the liquid rate. This provided a means of determining the deterioration of the packing due to liquid coalescence throughout the column.

His laboratory had built a series of columns, of 4 in., 6 in., 8 in., and 12 in. diam. up to approximately 12 ft. The absorption coefficient in such a system was defined as

$$K_G a = \frac{N_1 - N_2}{hA\ y_{1m}}.$$

For any given liquid rate $K_G a$ was constant and independent of the gas rate, and therefore N_1 and N_2 were functions of carbon dioxide concentration. This permitted a data analysis by plotting the ratio of log y_1/y_2 (where y_1 is the inlet concentration and y_2 the exit concentration of the carbon dioxide in the air) versus column heights, at constant liquid rates. For the ideal case this should produce straight lines of slopes characteristic of the liquid rate in the packing. If there was, however, a deterioration of liquid distribution somewhere in the tower then there should be expected a deviation from straight line behaviour, such that the slope decreased towards the bottom of the column. Furthermore the extent of the deviation could be used to ascertain what the new effective liquid rate was in the tower section. In order to provide data for such an analysis the columns were charged with increasing greater heights of packing and the functioning of the composite columns as height increased was examined.

These data provided the answer for the height to which a tower might be packed with any one packing in the laboratory-scale or pilot-scale column, before serious deterioration of performance would set in. It was reported that with $\frac{1}{2}$ in. rings in a 6 in. diam. column serious maldistribution occurred with only 3 ft. of packing whereas with saddles a height of 6 ft. could be reached. It was possible to express this limiting height as a function of the ratio of packing size to tube size. Professor Guyer and his co-workers at Zurich had used a similar analysis for presenting absorption data of ammonia in water.

Dr. H. R. C. **Pratt** said that it was necessary to consider two ranges of packing size, one below $\frac{1}{2}$ to $\frac{3}{4}$ in. where surface tension effects played a large part in wetting and small voids in the packing tended to be completely filled and stagnant, and larger sizes where the wetting was largely a film mechanism. If one considered only the larger packings and confined one's consideration to a single liquid, such as water or any organic liquid, and plotted curves of the apparent volume coefficient for the gas film against liquid rate at fixed values of the gas rate, curves similar to Fig. 1 would be obtained.[1]

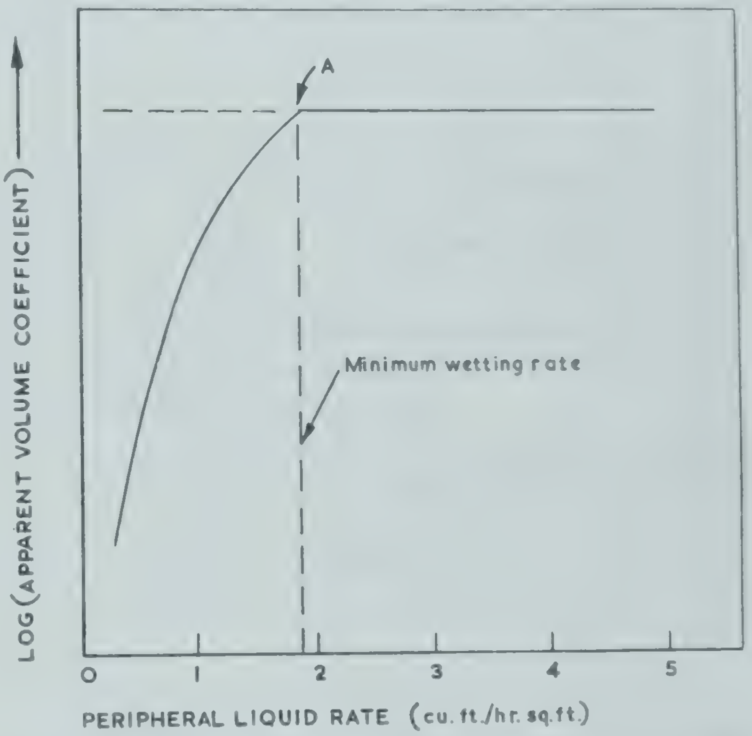


Fig. 1.—*Typical graph of apparent volume coefficient for gas-film controlled system against liquid-rate for fixed gas-rate*

The upper, almost horizontal, part corresponds with most regular (*e.g.* stacked ring and grid) packings to complete wetting of the packing, and with most random packings to the maximum possible degree of wetting. Below the point of inflexion, A, the coefficient falls off rapidly ; this appears to be due to a reduction in effective area, the volume coefficient being proportional to $K_g a .\ W_1 .\ W_2$ where K_g is the area coefficient (almost constant for constant gas rate), a, is the superficial area of the packing, W_1 a static wetting factor, such that aW_1 is the maximum superficial area at high liquid rates, and W_2 the dynamic wetting factor.

The value of W_2 is dependent, among other things, upon the ratio of the actual liquid rate to that at A, *i.e.* to the minimum liquid rate for complete or maximum wetting with the particular solute used.

Conditions are different for a liquid-controlled system, where liquid rates are normally higher and it is less usual to work below the point of maximum wetting. However, the available data show that there is no sharp break in the plot of apparent volume coefficient against liquid rate at the expected point of complete wetting. This appears to be due to the dependence of the liquid film resistance upon liquid rate, since if the degree of wetting diminishes, the liquid rate over the remaining wetted portion increases. Expressed algebraically,

$$K_l a \text{ (apparent)} = K_l \, a W_1 \, W_2$$

Also, $K_l = \text{const.} \times L^n$, or, if wetting is incomplete,

$$K_l = \text{const.} \times (L/W_2)^n,$$

$$\therefore K_l a \text{ (apparent)} = \text{const.} \times L^n a \; W_1 \; W_2^{(1-n)}$$

Hence, since the exponent n has a value of 0·7–0·9, the effect of incomplete wetting is to reduce the volume coefficient, extrapolated from liquid rates above the minimum wetting point, by a factor of $W_2^{0\cdot1-0\cdot3}$. This factor is so close to unity in most cases that the effect of incomplete wetting is usually indistinguishable for liquid-film controlled systems.

In a mixed system, with gas and liquid films both controlling the expression becomes :

$$\frac{1}{K_{og} \, a W_1 \, \mathrm{f}(W_2)} = \frac{1}{K_g \, a W_1 \, W_2} = \frac{H}{K_l \, a W_1 \, W_2^{(1-n)}}$$

where $\mathrm{f}(W_2)$ refers to the effect of incomplete wetting on the overall coefficient. Hence the analysis of performance data for such systems is rather difficult.

Returning to Mr. Hoftyzer's paper, Dr. Pratt thought that, if due allowances were made for these wetting effects, laboratory model test data for columns packed with ½ to ¾ in. rings could be scaled-up reasonably well to larger columns packed with rings of 1 in. to 2 in. or greater ; but in general he agreed with the author that the scaling-up was difficult if very small packings in laboratory size columns were used for the model tests.

Mr. Hoftyzer also mentioned in the case of bubble plates that a full-size bubble cap must be tested. Thus, it was possible to carry out a small-scale test with a column containing one full-scale cap per plate and then to scale-up to one with a large number of caps per plate. However, it would not appear unreasonable to use one single full-sized slot per plate, in a very small diameter column. Given the right ratio of cross-sectional area of slot to total cross-sectional area of column, it would then be possible to scale-up over a very much larger ratio of sizes.

Mr. T. A. KANTYKA said that the whole argument developed by Mr. Hoftyzer was based on the assumption that in scaling-up the dimensional similarity demanded by modified Reynolds number and Froude number must be satisfied simultaneously. There was such a variety of dimensionless groups associated with Dr. Reynolds in these days that one became a little confused, and Mr. Kantyka subscribed to the apprehensions expressed the previous day concerning the indiscriminate manipulation and use of dimensionless groups. To carry the author's argument to a logical conclusion, the condition of similarity ought to be extended to three dimensionless groups *viz.* : the Reynolds numbers based on the gas and liquid phases and the Froude number. To satisfy this set of conditions would be practically impossible.

The author's proof of his argument was limited to one particular example, the absorption of carbon dioxide by water, and therefore limited to the case where the liquid film coefficient was predominant. The fact that $k_L a$ was used in the correlation would suggest that the state of wetting was not properly known, and therefore one might wonder whether the packing was completely wetted, as had already been pointed out by Dr. Pratt.

It was an established practice[2] to correlate absorption data in terms of absorption coefficient based on a falling film column, and these coefficients were then corrected for the actual packing to be used by means of the so-called packing factors. The gas and liquid packing factors, R_g and R_l, for a range of stoneware random rings of diameter D, in. are given in Table I.

TABLE I.—*Gas and Liquid Packing Factors*

D (in.) .	3	2	1½	1	¾	½
R_g .	2·5	2·7	2·7	2·7	2·7	2·7
Rl .	0·6	0·65	0·75	0·88	0·88	0·88

The values were based on a considerable number of measurements on full-scale plant and referred to fully wetted conditions. The constancy of R_g showed clearly that the author's argument did not apply to cases where the absorption was gas film controlled. For the liquid film packing factor, there was some variation over the range of packing sizes but he suggested that these could be accounted for in conjunction with the problem of wetting by the changes in the flow pattern, by which he meant the differences in the distribution of residence times of liquid particles, which in a packed column by no means corresponded to that of a plug flow.

Dr. O. P. Kharbanda, referring to the paper by Hagger, Jones, and Pratt, asked what type of stainless steel had been used on the full-scale plant and what method of analysis for hydrogen sulphide had been employed.

Turning to the paper by Schuftan and Forrest, he expressed some disappointment at the small amount of information given by the authors.

Taking as an example the section on rectification columns, it was stated that packed columns were quite suitable for small plants. That was well known, but it would have been useful to know how small. The authors went on to say, "When scaling-up tray columns the vapour load is roughly proportional to the square of the diameter". In fact the reader was being told that the area was proportional to the square of the diameter! The authors went on to say that this would normally require an increase in the tray spacing, but that this was only possible to a limited extent as cold losses had to be kept at a minimum. That was interesting, but it would have been useful to have some facts and figures, particularly on the design features of the column.

Had the authors used perforated trays? If so, there were some factors which would not be scaled-up, but which might remain constant irrespective of the size of the column. The factors which he had in mind were the perforation area (expressed as a percentage of the tray area) and the perforation diameter. Those might be expected to remain substantially the same, but it would be interesting to know what the authors' experience had been.

Dr. L. L. Katan congratulated Messrs. Hagger, Jones, and Pratt on making one of the most courageous statements which he had ever seen in a paper, when they wrote (p. S78) "This paper is not put forward as an example of a scale-up based on academic chemical engineering principles, but rather as an example of how scaling-up often proceeds". That was something which many people had done, but how many would have the courage to admit it?

Could the authors explain how they came to decide to go to a spray tower, as opposed to a packed tower or some other type? They said on p. S80 "As a result of these experiments, and of other considerations, it was decided to develop spray chambers rather than packed towers". They also stated that an important factor in these considerations was the reduced resistance to the air stream, but elsewhere in the paper they referred to the power consumption of liquid pumping as being a most serious consideration. Again, under "Variables Considered" they described the changes in ratio of sodium carbonate to bicarbonate and referred to chemical composition as being an important factor, but elsewhere they said that the film coefficient was limiting.

He wondered whether the authors' choice of a horizontal "cave" might have an application for water cooling towers. Obviously where natural draught was what was wanted it would not apply, but forced or induced draught towers were common, and it would seem that this idea might have a valuable application there.

Mr. G. U. Hopton said with regard to the use of horizontal vessels for mass transfer and for heat transfer, that he was not aware of any application of the horizontal "cave" for cooling towers, but for gas cooling and similar operations it was sometimes very convenient to use instead of a vertical tower.

Mr. Hoftyzer's paper was of great interest. It had attracted a great deal of discussion, and he would advise the author to bear in mind the well-known Latin proverb, "Illegitimo non carborundum". The scaling-up of mass transfer equipment was, of course, extremely difficult, and Mr. Hopton was inclined to be even gloomier than Dr. Pratt. There was always some difficulty, however careful the academic or empirical treatment might be. There was a well-known law which chemical engineers should bear in mind called the Law of Inanimate Malice. It was so important that a famous professor once delivered a lecture on the subject before a learned body. He took a slice of bread, spread it thickly with butter and jam, held it up, and said "I will now demonstrate the Law of Inanimate Malice. I shall drop this on your beautiful carpet, and you will see what a mess it makes". Before the horrified members could stop him he dropped it—and, of course, it fell with the dry side on the carpet. That was a perfect example of the operation of the law.

There were two main aspects of scaling-up. There was first of all the engineering side—such matters as the distribution of the liquor on the packing. It was extremely difficult to get anything like really good distribution, whatever the type of packing used. So far as bubble cap towers were concerned, he was astonished to hear that it was possible to scale-up from one cap or one slot ; he would not dream of trying to do so. The nature of the flow on a bubble cap tray was very complex —whether it flowed across the diameter of the column, preferential paths and so on. Even if it were to flow evenly across, if the downcomers were on the opposite side there would be a big difference in the concentration gradient. Coming across the tray and then down there would be very little difference in the concentration of the liquor near the downcomer, whereas on the far side there would be the greatest possible difference in the concentration gradient, so that for vapours coming up more or less evenly distributed the effect would be very different over that tray. An attempt had been made to meet that by arranging to come always in the same sense down the column.

Those difficulties were well known, but there was another, and that was knowledge of the actual physico-chemical process. Not nearly enough was known about the mechanism from a physico-chemical standpoint. A short time ago he introduced two German films on mass transfer in liquids to The Institution of Chemical Engineers.[3, 4] They showed that when transferring a solute from a droplet into a continuous phase surrounding it the transfer was not a steady mechanism at all, as might be assumed from the two-film theory. It was shown clearly by the Schlieren technique to proceed in a violent manner, like a series of eruptions. It was that sort of knowledge which was not available, and until more was known there would be difficulties in scaling-up by whatever method.

Dr. F. V. A. Engel wrote :

In his conclusion, Dr. P. J. Hoftyzer mentions that results of the performance of the full-scale plant are only 25% of those of the pilot plant. There may be two reasons for this discrepancy.

First of all, it may be due to a so-called " scale effect " in which many incoherent influencing characteristics are lumped together. The process may be characterised by some incompatible laws of dynamic similarity. This means, that either one or another law which may have a considerable influence upon the process has been neglected and the scaling-up has been done only following one of the characteristic laws of dynamic similarity, say, for instance, the Reynolds law, whilst from a strict viewpoint of dynamic similarity the Froude law may have just the same deciding influence.

The second consideration, which may account for considerable discrepancies, is the choice of the various integrating quantities forming a specific model law of dynamic similarity. The latter point may be explained by the choice of the length dimension in a criterion which is a Reynolds number and which forms the basis of Fig. 1 of the author's paper. The choice of the length dimension may be physically correct, but it may neglect another aspect of the Reynolds number, namely the Reynolds number related to the diameter of the column. In a complex plant it may be necessary to characterise the process at least by two Reynolds numbers, one referring to the column diameter and the other " to the total surface area per unit volume " of the packing material. Of course, the Froude number may also be of deciding influence.

The author rightly remarks that more accurate prediction is not possible, as further data on liquid distribution is lacking. However, this may not be the only influencing factor. In my opinion " scale effects " should be studied more carefully. It is well known that in civil engineering scale ratios of 1 to 25, or even 1 to 40, may result in discrepancies between the model and the prototype of the order of 3 or 4% only. Partly this is due to the fact that the dimensions of those structures are very large in comparison to the crucial dimensions of chemical plants.

The discrepancies mentioned by the author are so large that it seems to be advisable to investigate " a family of models " covering an extensive range of scale ratios. Investigations on a single pilot plant and the assumption that the full-scale plant may show a dynamical similar behaviour seems not yet to have been corroborated. In view of the complex nature of chemical process plants the suggestion made in the introductory paper by Mr. Edgeworth Johnstone and Prof. Thring, that plant *elements* and models of those *elements* should be investigated, should also be carefully considered.

There is another point which I should like to raise, namely the presentation of a non-dimensional group on p. S74 of the author's paper, which is a particular version of the Weber number. In accordance with some axioms and laws of formation[5] the velocity term is an essential variable in a non-dimensional group related to the laws of dynamic similarity. Furthermore, it is in general not permissible to replace, say, the velocity by any other quantities or combination of such quantities which, by mere chance, may have the same dimension as a velocity or a square of a velocity.

Mr. Hoftyzer also includes, in the term which should be the Weber number, acceleration of gravity. This may be due to the fact that he gives the dimensions of surface tension in accordance with the Continental Technical System. However, as surface tension in the Technical System, as well as in the c.g.s.-system, is related to a force, most likely the introduction of g is due to the density term. Acceleration of gravity is a specific feature of the Froude number and should not occur in any other non-dimensional group. Non-dimensional groups should be presented in such a manner that they are applicable to any consistent system of measurement. Unfortunately the Continental Technical System is in some of its terms inconsistent, resulting in considerable confusion when dealing with non-dimensional groups. These confusions make it very cumbersome to study many engineering papers which present their test results on the basis of non-dimensional groups. A short time ago I therefore stressed the necessity of some international standards regarding non-dimensional groups forming the laws of dynamic similarity.[6]

Mr. James J. Priestley wrote :

A similar scale-up to the one described by Messrs. Hagger, Jones, and Pratt was carried out during the development of the "Manchester" liquid purification process for the removal of hydrogen sulphide from crude town gas. This is chemically the same as the "Ferrox Process", the main difference being the employment of multiple stage washing.

Data were obtained from a pilot plant. Generally speaking they were applicable to large contract plants subsequently installed. However, it was found that the influence of minor differences in gas composition was far greater than had been realised when working at the pilot-scale, and allowance had to be made for these differences, either in the construction or in the operation when the large plants actually went to work. Most marked was the influence of the hydrogen cyanide content, which only ranged from zero to less than 1000 p.p.m. With a gas containing no hydrogen cyanide all the purification had to be carried out using the ferric hydroxide/ferric sulphide cycle. Up to 500 p.p.m. the influence of hydrogen cyanide was negligible but at higher values the added degree of purification increased in proportion to the hydrogen cyanide content, due to the effect of complex iron cyanides acting as very efficient catalysts. Indeed the use of these cyanides or "blues" is the basis of the Staatsmijnen-Otto process, described by Pieters and Van Krevelen.[7] Another trace substance, not identified, but probably a phenolic oil, was found to have a profound effect on frothing in oxidisers. These effects were not, and moreover could not, have been forecast with any accuracy from pilot plant work on gas from a single works.

Blocking of board filling was overcome in the pilot plant towers which were 3 ft. dia. by replacing it with "disc and doughnut" type filling. When towers 8 ft. 6 in. dia. had to be used on the contract plants an alternative idea had to be found, as the wetted periphery only increases in proportion to the diameter and the allowable gas flow (at a given velocity) to the square of the diameter. This was subsequently overcome by using "splash plates"—a layer of narrow flat horizontal plates with a rectangular space between them, each layer staggered in relation to its neighbours. above and below. Here the wetted periphery could be any chosen length, within limits; a

Table II.—*Comparison of Courtaulds Plant and Holmes Plant*

	Courtaulds	W. C. Holmes "Manchester" Plant at Bristol
Gas volume (cu. ft./hr.)	7 million	250 000
Hydrogen sulphide concn. inlet	300 p.p.m.	10 500 p.p.m.
Hydrogen sulphide concn. outlet	10/20 p.p.m.	0·3 p.p.m.
Absorbers . .	One sprayed horizontal scrubber	5 towers in series : 2 @ 8 ft. 6 in. diam. ; 3 @ 10 ft. square
Alkali . . .	NaOH	$Na_2CO_3/NaHCO_3$
Absorber filling .	Sprays only	" Splash plates "
K_1 . . .	3·0–5·0	1·2 (No. 1 washer) 1·37 (No. 2 washer)
K_2 . . .	0·084–0·15	0·146
Liquor flow (gal./hr/cu. ft. absorber)	25–48	82·0
Liquor flow (gal./1000 cu. ft. gas)	1·23	960·0
Iron catalyst in suspension	1·0%	0·01%

scale-up using the same principles but a different physical arrangement.

The authors may be interested in a comparison of their own plant with the "Manchester" liquid purification plant installed at the Stapleton Road Gas Works, Bristol. Comparative details are given in Table II.

The *weight* of iron-catalyst is about the same when expressed as pounds per 1000 cu. ft. of gas. The lower concentration in the Holmes plant is inevitable as iron is continually frothed off during the oxidation stage. We can confirm the improvement in removal efficiency with the increase in alkalinity. Because of the stringent limit placed on the outlet hydrogen sulphide concentration by the Gas Referees, it has been our practice to feed the fresh sodium carbonate required for the process into the last absorption tower to take advantage of this. On the other hand the rate of oxidation of the catalyst in the reagent preparation tank decreases with increase in alkalinity and is at its maximum in a neutral solution.

Authors' Replies

Mr. P. J. Hoftyzer said in reply to Mr. Andrews that absorption combined with chemical reaction was quite different from that given as an example in his paper, *i.e.* the absorption of carbon dioxide by water. Reference had been made to the absorption of carbon dioxide in caustic solutions, and Mr. Hoftyzer agreed that if in this case the chemical reaction was determining, then the absorption rate per unit of interfacial area was equal in any type of apparatus. But even in this case the rate of physical mass transfer in the liquid phase should be known in both kinds of apparatus, to be sure that the chemical reaction rate was determining in both cases.

The second point concerned equation (3) p. S76 and the dependence of the coefficient c on the diameter of the packing. The equation he had used was not the equation published by van Krevelen and van Hooren, but was a simplified version of it. What Mr. Andrews had done in plotting the coefficient c against the diameter of the packing (D) was what Mr. Hoftyzer had done. If the coefficient c was plotted against the diameter of the packing for the laboratory scale and the semi-technical scale experiments, most of the results on the technical scale would lie to the right of the curve in the shaded area (see Fig. 2). That was why he could distinguish between the effects of the diameter of the packing on the one side and of the liquid distribution on the other.

Mr. Hoftyzer agreed that a better correlation could be obtained if D (or $1/a_p$) was eliminated from the first group of equation (3), as in the equation proposed by Mr. Andrews. In fact, an equation of this type had been used by van Krevelen and himself in 1948.[8] Such an equation could be derived from equation (3), by combining D or a_p with other quantities to give a dimensionless number. In this case μ, ρ, and g were used, but a similar result could be obtained by using σ instead of μ. As water was the absorption liquid in all the experiments compared, however, the validity of the equation could not be checked. As long as the full effect of the physical properties is not known, it will not be possible to evaluate the proper equation.

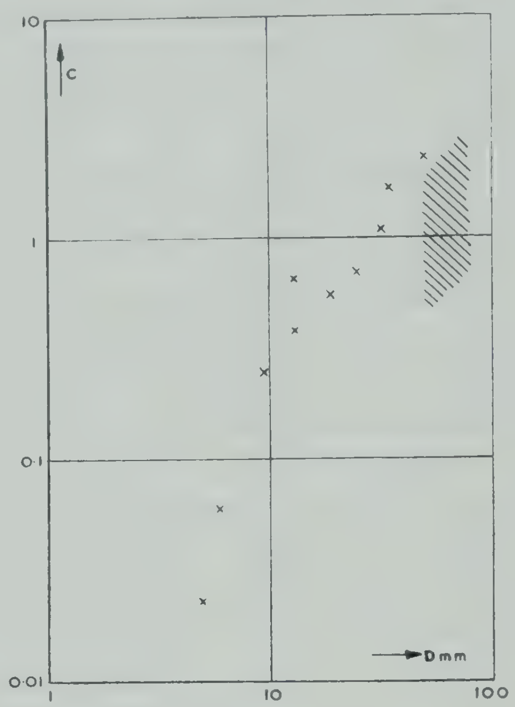


Fig. 2

The third, and important, point, was that the effect of liquid distribution on the overall absorption coefficient of the tower depended on the system used and on the number of transfer units in the column. The larger the number of transfer units, the larger the effect of liquid distribution on the absorption coefficient. Mr. Hoftyzer agreed that the uneven distribution of liquid could be detected by using systems with a large number of transfer units, but it could be found even better by doing experiments on selective absorption, as for instance by trying to absorb hydrogen sulphide by caustic solutions from a mixture containing carbon dioxide.

The experiments mentioned by Mr. Leva were interesting. They were a contribution to our knowledge about liquid distribution. As they dealt with the absorption of carbon dioxide in caustic solution, it could be expected that the absorption rate per unit of wetted surface area would be equal anywhere, independent of the local liquid rate. Therefore the total wetted area was measured and the results obtained with this system could not be applied to cases in which the rate of physical mass transfer in the liquid was determining.

He agreed with Dr. Pratt that surface tension was the important factor for the smaller rings. He did not suppose, however, that the discrepancies between laboratory experiments and technical data could be eliminated by taking into account the minimum wetting point, as both groups of data had been obtained at liquid rates higher than the minimum point.

Experiments with models of plate columns consisting of only one slot could be useful, if the influence of gas streams issuing from separated slots on each other could be neglected ; if not, the model should at least contain one bubble cap.

Mr. Hoftyzer later wrote in reply to Mr. Kantyka :

I should like to give some commentary on Mr. Kantyka's remarks because these evince a tendency to simplification. For example, the experimentally determined value of k_La for a given column is considered as the product of a transfer coefficient, k_L, which holds for the entire column and the specific surface area, a. If the packing is in a fully wetted condition, a is known and k_L can be derived. In reality k_L varies from point to point through the column depending on the liquid distribution. As a result the average value of k_L thus calculated depends on all sorts of quantities, such as the ratio of H/D for the column (mentioned by Mr. Leva) and the number of transfer units (mentioned by Mr. Andrew).

Furthermore, many of the empirical formulae for calculating the transfer coefficient in a packed column are over-simplified. Through lack of good calculation methods the designer is forced to use an empirical formula. However, he should not labour under the illusion that more than the order of magnitude of the transfer coefficients can be determined in this way.

For example, in Morris and Jackson's formula for k_L, which was cited by Mr. Kantyka, all sorts of factors have been left out. It is to be regretted therefore that the considerable number of measurements on full-scale plant which, according to Mr. Kantyka, should confirm the correctness of this formula has not been published as yet.

Mr. Hoftyzer later wrote in reply to Dr. Engel :

Dr. Engel mentions two causes that are responsible for the discrepancy between the results obtained with pilot and full-scale plants. His first suggestion, which ascribes the discrepancy to a so-called scale-effect, is a trivial one ; the second is incorrect. The discrepancy is caused not by the comparison being based on ill-chosen non-dimensional quantities but by an unequal distribution of the liquid through the column. The flow pattern can never be completely described by quantities which only include the flowrate averaged over the entire column.

It is difficult to see Dr. Engel's objection to the use of the non-dimensional group $\sigma/g\rho D^2$. In the application of the dimension analysis there is not a single law which forbids the formation of this quantity. Its physical significance is a comparison between the effects of surface tension and gravity and that is exactly the point at issue here. For a very lucid exposition on the derivation of non-dimensional quantities reference may be made to the article by Klinkenberg and Mooy.[9]

In his remarks on the use of units Dr. Engel confounds the properties of dynamic and static unit systems. All quantities used in the present paper are expressed in terms of the dynamic M.K.S. system, where the surface tension has the dimension $M.T^{-2}$. The physical significance of the acceleration of gravity g in the above non-dimensional quantity has already been explained. If the surface tension were expressed in terms of a static system and the density were replaced by the specific gravity, the factor g would no longer occur in the non-dimensional quantity.

This is a drawback of using a static system.

Mr. D. C. F. Pratt, replying, said that not very much stainless steel was used in the absorption plant described in his paper ; most of the units were constructed of mild steel with a bitumen lining. The gas entering the scrubbing units contained sulphuric acid and various salts in suspension, and was corrosive. Once it had entered the chamber in which it was scrubbed with the alkaline liquor, the suspended droplets were neutralised, but even then the gas was corrosive. There were some filter screens made from 18/8 stainless steel for the alkaline scrubbing liquor. These screens were resistant but mild steel ones rapidly corroded. At the outlet of the chamber the gas carried a lot of liquid droplets and it was necessary to have an eliminator to get rid of them. This was made of 18/8 steel.

For the analysis of hydrogen sulphide a continuous meter was being developed, but it had not yet been finished. All analysis had been done by normal sampling methods, taking a known volume of gas and bubbling through a cadmium acetate solution, giving cadmium sulphide, which was titrated with iodine. It was necessary to be careful about the effects of sunlight ; if the sample bottles were left in bright light the cadmium sulphide could oxidise and the answer would be wrong. Sampling had been found to be unusually difficult. The ducts were big, about 15 ft. square, so that unless the correct procedure of sampling at various points across the duct was carried out there were obvious difficulties through getting layers of different concentrations. That trouble might be expected, because of scale effects ; but the trouble that they did not expect, but which occurred, was due to leaking sample lines. At one time it was thought that one of the units was scrubbing down to 3 p.p.m., but actually the figure was 40. That had been merely because the sample lines had been under a negative pressure and leaked somewhere. It was possible to go on getting the wrong answer for months unless one checked and re-checked and duplicated the sample lines.

The authors were very grateful for Dr. Katan's remarks. The reason for changing to spray towers was not very clear from the paper, but some of the previous experience to which they referred, when glass plate packing had been used, had not been very good. The pilot-plant had initially been operated with packing, but it was found that the unit worked better with the packing removed. Because the unit was operated with an iron oxide slurry as scrubbing liquor, sulphur came out of solution. Solids were found to build up on the plates. It had been largely for that reason that it had been decided to change to spray towers.

With regard to the input power, they had not been concerned so much about the actual power required as about obtaining fans to handle the load. At the time they had not thought that they could get a fan to handle the very large flows with a higher pressure drop. They believed that they could now get such fans, but they had been dealing with an existing system and existing fans, and they felt that they must restrict the pressure drop to 2 in. w.g. In fact it was 1 in.

There was no contradiction with regard to the sodium carbonate and sodium bicarbonate. The point was that the carbonate/bicarbonate ratio had an effect if something went wrong.

Horizontal chambers had many advantages in both capital and running costs and were often used in air-conditioning units where the air went horizontally through the sprays. In various acetone absorption plants at Courtaulds the method was used, and it might well have an application for forced-draught cooling towers.

The authors thanked Mr. Priestley for his written contribution and found the comparative figures very interesting. In Mr. Priestley's Table II, however, they pointed out that although sodium hydroxide was used as make-up to the Courtaulds plant, the scrubbing liquor was nevertheless sodium carbonate and sodium bicarbonate as in the Holmes plant, since carbon dioxide was absorbed from the air.

Dr. P. M. SCHUFTAN said that he and his co-author were sorry to have disappointed Dr. Kharbanda by not giving sufficient data. The main problems did not lie on the chemical engineering side but in other directions—operational, mechanical, and economic. He did not believe that there had been any deviation on their part from the established rule and chemical engineering practice.

The heat exchangers could be designed by ordinary methods, and no fouling was likely to occur.

The limiting size of a packed column was about 6 in.

Taken out of its context, the statement in the paper that " the vapour load is roughly proportional to the square of the diameter " was, of course, a platitude, but the emphasis was on the next part of the sentence, where it was stated that the weir loads were much larger, and special mechanical provisions had to be made in order to remove the liquid.

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EXPERIMENTS WITH FURNACE MODELS

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SUMMARY

Flow patterns in furnaces have been investigated by the use of small-scale models. During the first stage the models were of gas-fired open-hearth furnaces, water being used as flow medium. Techniques were devised to give good flow visualisation, the main object being the construction of a complete picture of flow pattern.

More recently, the emphasis has been on the use of models in which air is the flow medium. Visualisation is more difficult but quantitative work is much easier than when using water.

Equality of Reynolds number in model and prototype is taken as the main criterion of similarity, the models generally being geometrically similar to their respective prototypes. For oil-fired furnaces, however, it is necessary to distort the simple geometric scaling-down of the burner nozzle. This is important because the momentum of the fuel stream entering the furnace is the greatest single factor affecting the flow pattern in the furnace chamber.

Examples of the use of models in dealing with problems of roof wear, slag deposition, and temperature distribution are given.

Introduction

An important feature governing the behaviour of a metallurgical furnace is the flow pattern of the combustion gases. The first account of the use of models in a study of furnace flow patterns is that by Groume-Grjimailo,[1] published in 1911. The importance of flow patterns was particularly appreciated by the United Steel Companies after an extended series of trials[2] on open-hearth steelmaking furnaces which was completed in 1946. A programme of water-model work was commenced in an attempt to learn something of the significance of the patterns and the way in which they could be modified. A considerable expenditure of effort was required at first to develop suitable visualisation and photographic techniques. The results of the first stage of the work are described in detail in publications made in 1949.[3]

Initially, there was doubt as to the validity of the results obtained with water models since the effects of buoyancy and rapid expansion due to combustion were not included. However, a considerable amount of work has been done to determine the flow pattern in full-scale furnaces. Within the limits of accuracy of the methods available, this work[4, 5] has confirmed the conclusion that it is possible to reproduce open-hearth furnace flow patterns with water and air models.

Following this first stage, which established the main features of the flow pattern, the later work has been of a more detailed nature, and has enabled problems such as roof wear and the effects of burner position to be investigated.

A wide variety of problems encountered on other types of furnaces has also been dealt with. Two such problems are described in this paper.

There has been an extensive development in the use of models throughout the glass and steel industries, particularly during the last ten years. This has included the use of small-scale models with either water or air as flow medium,[6] small-scale hot models[7] in which problems of combustion and heat transfer have been studied and work on full-scale plant through which cold air has been passed,[8] in some instances the flow pattern being established by observers inside the furnaces.[9] No attempt has been made in this paper to review this whole field. Emphasis has been put on a consideration of the similarity criteria involved, since it is this upon which the final design of a model depends if results of most value are to be obtained.

Similarity in Open-hearth Furnace Models

Four cases are considered. These arise from the two types of furnaces used (gas or oil-fired), and the choice of either of two fluids to be used in the cold model (water or air).

When calculating the necessary water or air flow rates to be used in the model it is necessary to specify a temperature which is representative

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of conditions in the prototype. In the open-hearth furnace, gas temperatures vary at any one time from about 1100° C. to probably above 1800° C. In such conditions, the selection of temperature must depend partly upon the section of the furnace in which the investigator is interested. The safest approach then is to operate the model over the range of Reynolds numbers calculated from reference to the temperature range. Generally the conditions are so turbulent that the flow pattern is insensitive to quite wide variations in Reynolds number.

Gas-fired furnaces

The important feature of these furnaces is that gaseous fuel and air enter the furnace from their respective ports at atmospheric pressure and with densities of a similar order of magnitude to the rest of the gases in the furnace. For this reason, models are used which are, in all respects, geometrically similar to the prototype.

WATER MODELS

The basis for similarity is that of equality of Reynolds number in the furnace and model. The Sutherland formula[10] is used to calculate the viscosity of the furnace gases which is assumed to be equal to that of air at the same temperature.

In earlier work[3] it was usual to make this calculation for the conditions in the furnace uptake, using the temperature of the preheated combustion air. In more recent work it has been usual to use a gas temperature which is representative of the conditions in the furnace chamber.

The quantity of water required to represent the air flow having been determined, the water flow to represent the producer gas is calculated by using the same mass ratio as for the air : gas mass ratio in the furnace. Under the conditions assumed in the furnace, producer gas and air have equal densities so that equal mass ratio also gives equal volume ratio.

To illustrate this point, typical figures for a 1/24-scale model are shown in Table I.

For these conditions, events occur in the water model 2·5 times faster than in the prototype. This comparatively small amount of speeding up, combined with the use of suitable tracers, makes visualisation in water models relatively easy. The tracer now used in water models is granular polystyrene of size range − 22 to + 44 B.S.S. This has a good surface reflectivity and a specific gravity of 1·023.

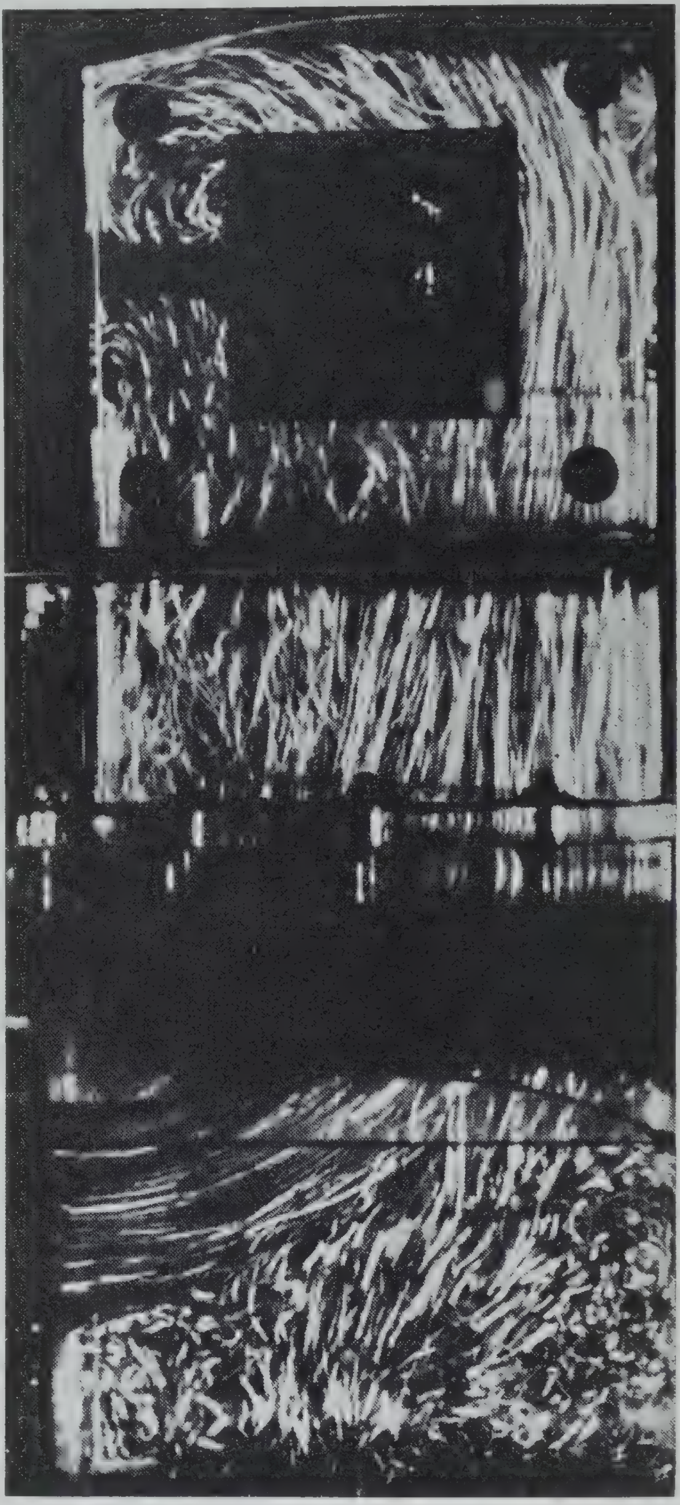


Fig. 1.—*Flow pattern in the uptake of a single uptake open-hearth furnace. Water model with polystrene tracer*

Table I.—*Typical Values of Flow Rates for* 1/24-*scale Furnace Model*

	Furnace	Water model	Air model
Producer gas	247 000 N. cu. ft./hr.	1450 gal. per hr.	2960 N. cu. ft./hr.
Air flow	313 000 N. cu. ft./hr.	2100 gal. per hr.	4300 N. cu. ft./hr.
Area of two air uptakes	14·2 sq. ft.		
Area of gas port	2·7 sq. ft.		
Velocity :			
Air uptake	36·4 ft./sec.	3·79 ft./sec.	55·5 ft./sec.
Gas port	132 ft./sec.	13·8 ft./sec.	201 ft./sec.

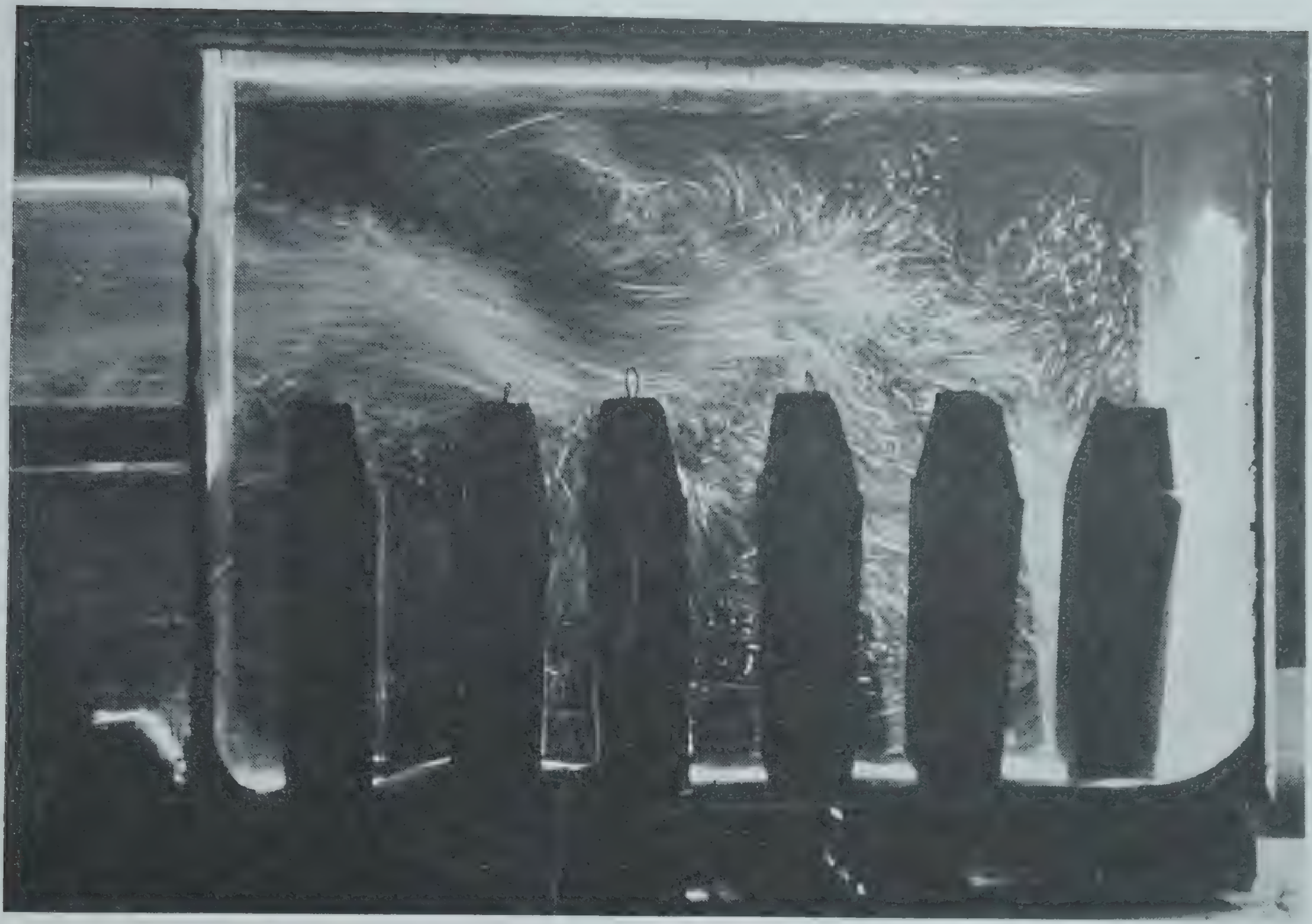


Fig. 2.—*Flow pattern in an air model of a soaking pit with balsa dust as tracer. This shows short-circuiting of some of the combustion gases. The gas offtake is below the burner port*

AIR MODELS

Equality of Reynolds number in model and prototype is again used as the criterion of similarity.

For the case already considered, the values of air flow required are shown in Table I. If the time scales are compared when using an air model for these conditions, it is found that events occur 36·6 times faster in the model than in the furnace. It is because of this considerable speeding up in the air model that it is generally more difficult to appreciate details of flow pattern by direct visual examination. This disadvantage may be overcome by using a camera to record the pattern at any instant. Even then, however, it is less easy to obtain good photographic results with an air model. A tracer material commonly used for visualisation is balsa dust. Results obtained photographically in a water model and in an air model may be compared by examining Figs. 1 and 2.

Oil-fired furnaces

The chief feature of an oil-fired furnace, from considerations of similarity, is that liquid fuel and steam are injected into the furnace in a high velocity jet having a density about eight times greater than that of the flame gases. Because of this density difference the straightforward Reynolds analogy is inadequate. In a model, the density of the fluid leaving the burner is equal to, or approximately equal to, that of the fluid in the furnace model chamber. To allow for this difference in conditions between model and prototype the size of nozzle used in the model is larger than that obtained by simple scaling-down.

It is important that the correct nozzle size is used because any errors will seriously affect the flow pattern obtained. The conditions which it is necessary to fulfil have been formulated by Thring and Newby.[11]

WATER MODELS

The method consists essentially of determining the outlet size of the nozzle that would be required in the prototype to suit certain hypothetical conditions. For this purpose it is assumed that the steam/oil mixture leaves the nozzle in the furnace at the same mass flow rate as under actual conditions but with a density, pressure, and temperature equal to that of the flame gases. Further, this new nozzle size is such that the rate of production of momentum is the same in the hypothetical as in the actual case. This hypothetical nozzle diameter has been referred to as the " equivalent nozzle diameter ".[12]

The argument is that the actual and hypothetical prototypes would have similar flow and mixing patterns because, in the actual furnace, the high-density steam/oil mixture will, within a short distance of the nozzle, attain a density equal to that of the flame and still retain its initial momentum.

If the flame is now assumed to have a uniform temperature and density, it will entrain gases from its surroundings at a rate which is independent of the density of the air or gases being entrained [11].

In other words, within the limits of the assumptions made, the actual prototype will have the same mixing and flow patterns as the hypothetical prototype in which the density is constant throughout and the temperature constant at, say, 1700° C. This being so it is now only necessary to scale down the hypothetical prototype.

The ability to determine the nozzle size required in the hypothetical prototype under " equal density " conditions depends on a knowledge of conditions in the nozzle of the actual burner and the type of nozzle used, *i.e.* simple convergent or venturi-type. The simplest method of obtaining this information seems to be by making a direct measurement of the reaction of the burner to the jet, usually referred to as burner thrust. This has been done on test rigs in which full-scale open-hearth burners have been tested.[13, 14] Burner thrust has been measured for a variety of nozzle shapes and various oil/steam ratios.

The outlet diameter of the nozzle required in the equal density system may then be determined from the following simple relationships :—

$$G = \frac{M_n \cdot V_{en}}{g} \quad . \quad . \quad (1)$$

The mass flow rate of the nozzle fluid is known and, as the " equal density " system is being considered, the density of the steam/oil mixture is equal to that of the gases in the furnace.

Therefore, the velocity in the nozzle is :—

$$V_{en} = \frac{M_n}{\rho_f \, A_{en}} \quad . \quad . \quad (2)$$

Thus, nozzle size may be determined from equations (1) and (2)

$$A_{en} = \frac{M_n^{\,2}}{G \cdot g \cdot \rho_f} \quad . \quad . \quad (3)$$

Introducing the subscripts m and p to distinguish model and prototype, the nozzle size required in the water model is :—

$$D_{em} = D_{ep} \cdot L_m/L_p \quad . \quad . \quad (4)$$

This nozzle diameter is independent of the fraction of correct Reynolds number at which the model is run.

It is interesting to compare the actual values of burner thrust obtained in the model and prototype.

Thrust of the model burner (all terms referring to nozzle conditions) is :—

$$G_m \cdot g = A_m \cdot \rho_m \cdot V_m^{\,2}$$

By substituting for A_m in terms of the equivalent nozzle diameter, it may be shown that :—

$$G_m = G_p \cdot \left(\frac{\mu_m}{\mu_p}\right)^2 \cdot \left(\frac{\rho_p}{\rho_m}\right) \quad . \quad (5)$$

The density in the prototype is taken as being equal to that of the flame.

Thus, the thrust of the model burner is independent of model scale and depends only on the viscosity and density of the fluid used. The same result may be obtained by starting with the generalised dimensionless equation for fluid flow derived in the introductory paper of this conference.[15]

AIR MODELS

The method adopted for a water model is only adequate for an air model if the air leaving the nozzle of the burner in the model has a pressure and a temperature equal to the model furnace chamber conditions. In this case the nozzle size is the same as in the water model. Generally, however, this condition is not fulfilled. Air temperature may be lower, due to adiabatic cooling during expansion through the nozzle if cold air is supplied to the burner, or higher if hot air is supplied to the burner as in one of the methods of obtaining mixing patterns. In addition to a possible temperature difference, the static pressure of the air leaving the nozzle will be above that of the surrounding air since it is usual to operate with sonic conditions in the model burner. This is due to the necessity of satisfying the calculated mass flow and thrust requirements.

For these reasons the "equivalent size", as determined for the water model, must be corrected.

There are a number of ways of calculating the required nozzle diameter. The method outlined below seems to be the most direct. The thrust, or jet reaction, from a convergent nozzle is given by :—

$$G = (P_n - P_a) \, . \, A_n + (M_n \, . \, V_n)/g \quad . \qquad (6)$$

but

$$A_n = M_n/(V_n \rho_n) \text{ and } P_n = \rho_n \, . \, R \, . \, T_n$$

Substituting in equation (6) for $P_n \, . \, A_n$

$$P_a \, . \, A_n = \frac{M_n \, . \, V_n}{g} \left\{ 1 + \frac{R \, . \, T_n \, . \, g}{V_n^2} \right\} - G$$

As there are sonic conditions in the nozzle throat :—[16]

$$V_n = (\gamma R \, . \, T_n \, . \, g)^{\frac{1}{2}}$$

Thus :—

$$P_a \, . \, A_n = \frac{M_n \, . \, V_n}{g} \left(\frac{\gamma + 1}{\gamma} \right) - G \quad . \qquad (7)$$

Also, assuming that there is no heat loss or gain from the nozzle and that the air velocity upstream of the nozzle is negligible, the heat drop during expansion is :—

$$C_p \, (T_i - T_n) = \frac{V_n^2}{2 \, . \, g \, . \, J} = \frac{\gamma \, . \, R \, . \, T_n}{2J}$$

giving

$$T_n = T_i \, . \, \frac{2 \, . \, J \, . \, C_p}{\gamma R + 2J \, . \, C_p}$$

But

$$\frac{R}{J} = C_p - C_v = C_p \left(1 - \frac{1}{\gamma} \right)$$

so that

$$T_n = T_i \left(\frac{2}{\gamma + 1} \right) = 0{\cdot}833 \, T_i$$

and

$$V_n = \left(\frac{2}{\gamma + 1} \, . \, R \, . \, g \, . \, T_i \right)^{\frac{1}{2}} = 60{\cdot}06 \, T_i^{\frac{1}{2}}.$$

Substituting for V_n in equation (7), the nozzle area is :—

$$A_n = \frac{M_n}{P_a . g} \, . \left[2 \left(\frac{\gamma + 1}{\gamma} \right) . R . g . T_i \right]^{\frac{1}{2}} - \frac{G}{P_a} \quad . \qquad (8)$$

Assuming that the model is run at atmospheric pressure, values may be inserted in equation (8) for P_a and for the constants g, γ, and R, giving the final expression for nozzle area (in square inches) :—

$$A_n = 0{\cdot}218 \, . \, M_n \, . \, T_i^{\frac{1}{2}} - 0{\cdot}0680 \, G \quad . \qquad (9)$$

It will be seen that the nozzle in the model is independent of the efficiency of the nozzle. This is a valuable practical advantage, since the efficiency of any particular nozzle may not be known and, consequently, the nozzle profile is not important.

Nozzle efficiency is defined as :—

$$\eta_n = \frac{\text{Actual heat drop during expansion from } P_i \text{ to } P_n}{\text{Heat drop obtained with adiabatic expansion from } P_i \text{ to } P_n} \times 100\%.$$

A_n having been determined, P_n may be calculated from equation (6). If it is necessary to calculate P_i, the delivery pressure of the air behind the nozzle, then it is necessary to know, approximately at least, nozzle efficiency. It is suggested that for general purposes, and using simple convergent nozzles, an efficiency of 90% be used.[7]

An example is shown below. An air temperature of 270° C. is used because, for the case considered, mixing patterns are obtained from temperature measurements.

Furnace conditions

	lb./hr.	lb./sec.
Oil flow, 375 gal. per hr. . . .	3565	
Steam flow—7 lb./gal. of oil . . .	2625	
Total nozzle flow	6190	1·72

	lb./hr.	lb./sec.
Air flow : 16·1 lb./lb. of oil . . .	57 400	
Total mass flow through furnace . .	63 590	17·66
Burner thrust	80 lb. wt.	

Assumed conditions

Average temperature in furnace : 1700° C.
Average temperature in model : 54° C.
This is the calculated final mixture temperature.
Density in furnace : ·0112 lb./cu. ft.
Density in model, at 54° C. : ·0674 lb./cu. ft.
Viscosity of furnace gases at 1700° C. : $42{\cdot}1 \times 10^{-6}$ lb./ft. sec. Assumed equal to that of air at 1700° C.
Viscosity of air in model at 54° C. : $13{\cdot}2 \times 10^{-6}$ lb./ft. sec.
Absolute pressure in model and furnace : 14·7 lb./sq. in.
Temperature of air supplied to model burner : 270° C.
Temperature of air supplied to model as " combustion air " 40° C.
Model scale 1/24th of full-scale.

For equality of Reynolds numbers in the model and prototype furnace chambers, the mass flow is calculated using :—

$$M_m = M_p \, . \left(\frac{L_m}{L_p} \right) . \left(\frac{\mu_m}{\mu_p} \right) \quad . \qquad (10)$$

This gives a total flow through the model of 0·230 lb./sec. Maintaining the ratio of burner mass flow : total mass flow, the nozzle flow in the model is :—

$$0\cdot230 \times (6190)/(63590) = \cdot0223 \text{ lb./sec.}$$

The combustion air flow is represented by a flow in the model of

$$\begin{aligned} 0\cdot230 - \cdot022 &= \cdot208 \text{ lb./ sec.} \\ &= 9280 \text{ N. cu. ft./hr.} \end{aligned}$$

The thrust required in the model, using equation (5), is 1·31 lb. wt.

The nozzle area is now obtained by inserting the calculated values of mass flow and thrust, together with the air supply temperature, in equation (9). For this case the area required is ·0242 sq. in., *i.e.* 0·176 in. diameter.

The nozzle temperature is, as shown above, $0\cdot833\ T_i = 452°$ K.

Air velocity in the nozzle is $60\cdot06\ T_i^{\frac{1}{2}} = 1410$ ft./sec.

The nozzle pressure may now be calculated using equation (6), from which $P_n = 28\cdot5$ lb./sq. in. abs. The nozzle conditions in the model having been obtained, it is possible to check back to the nozzle area in the model for the " equal density " condition.

Thus, the equivalent nozzle area in the furnace is, by equation (3)

$$A_{ep} = \frac{1\cdot72^2}{80 \times 32\cdot2 \times \cdot0112} = 0\cdot102 \text{ sq. ft.}$$

and the equivalent diameter = 4·34 in.

Thus the equivalent area in the model, multiplying by $(L_m/L_p)^2$, is ·0256 sq. in., or 0·181 in. dia. This is the size of nozzle required, for the stated furnace conditions, in the water model.

In the " equal density " case, no compressibility effects :—

$$G_m = \frac{M_m \cdot V_{em}}{g}$$

$$\therefore \quad V_{em} = \frac{1\cdot30 \times 32\cdot2}{\cdot0221} = 1893 \text{ ft./sec.}$$

The actual nozzle area already obtained may now be converted, as a check, to the hypothetical " equal density conditions ". Thus :—

$$\begin{aligned} A_{em} &= A_n \times \frac{V_n}{V_{em}} \cdot \frac{P_n}{P_a} \cdot \frac{T_a}{T_n} \\ &= \cdot0256 \text{ sq. in.} \end{aligned}$$

This agrees with the value found previously.

If nozzle delivery pressure P_i, is required, a nozzle efficiency of 90% may be assumed.[7] Under these conditions, the temperature drop obtained of $(1 - 0\cdot833)\ T_i$, is 90% of the drop that would be obtained, for the same pressure ratio, P_i/P_n, under adiabatic conditions.

$$\text{Adiabatic temperature drop} = \frac{(1 - 0\cdot833)}{0\cdot9}\ T_i$$

$$\therefore \quad t_n/T_i = 0\cdot812$$

so that :—

$$P_n/P_i = (0\cdot812)^{\gamma/(\gamma-1)} = \cdot483$$

Thus delivery pressure required is

$$28\cdot5/\cdot483 = 59\cdot0 \text{ lb./sq. in. abs.}$$

Considering equation (9) again, it is clear that there are limits to the value of burner thrust that may be simulated in the model. For the example above, the theoretical limiting value of thrust obtainable in the model is, by putting $A_n = 0$:—

$$G_m = \frac{0\cdot218\ .\ M_n\ T_i^{\frac{1}{2}}}{0\cdot0680} = 1\cdot67 \text{ lb. wt.}$$

Using equation (5), this corresponds to a thrust in the prototype of 102 lb. wt.

The limit to the value of thrust that it is possible to simulate is set, in practice, by available compressor air pressure. Taking 110 lb./sq. in. abs. as a typical value, using equation (6) and remembering that $P_n\ .\ A_n$ is constant for specified conditions, the thrust in the model is 1·46 lb. wt. This is the equivalent of 90 lb. wt. thrust in the furnace.

If it becomes necessary to simulate conditions with a greater thrust then, after adopting the maximum values of air supply pressure and temperature, P_i and T_i, that are convenient, the model must be run with a lower Reynolds number than the prototype or use made of a model of larger scale. With the Reynolds number in the model reduced to a fraction, S, of that in furnace, it may be shown that the thrust required in the model is given by :—

$$G_m = G_p\ .\ (\mu_m/\mu_p)^2\ .\ (\rho_p/\rho_m)^2\ .\ S^2 \qquad . \qquad (10)$$

As already noted above, the equivalent nozzle size is unchanged. This is of particular convenience in water models.

If a hot model is constructed, in which fuel is burned to give the same temperature as in the prototype, then the burner thrust is the same in model and prototype. It may not be possible to obtain this condition because of the high pressure required to obtain the same thrust as in the prototype with a lower mass flow rate. The model would then have to be run with reduced mass flows, the correct ratio between air and burner fluid being maintained.

The use in model work of a nozzle the size of which has been determined by calculation has been criticised by Cashmore,[17] who prefers to make a direct measurement of model burner thrust. In the calculation of nozzle size it is assumed, equation (6), that velocity is uniform over the whole cross-section of the nozzle throat. This assumption is most in error if the velocity distribution is parabolic, as in streamline flow. In this case the actual value of the momentum corresponding to the term MV/g in equation (6) is 133% of the calculated value. If a velocity distribution is assumed which corresponds approximately to turbulent pipe flow, then the actual momentum is about 110% of the calculated value. This would give a total thrust of about 107% of the calculated figure. The mixing lengths obtained would then be 4% too short. This leads to the conclusion that calculated nozzle sizes are not likely to lead to gross errors but that, as further experimental data are obtained, the present weaknesses in this step in model design will be eliminated.

Use of Water Models

Water models have been used to establish the general flow pattern existing in various designs of open-hearth furnace, including the regenerators and the slag pockets. Water models are well suited to this purpose since visualisation is relatively easy and direct observation is usually sufficient to detect main features or the changes resulting from detailed alterations to parts of the model. The results of detailed flow pattern surveys have already been published elsewhere.[3] Fig. 3 shows a typical general pattern.

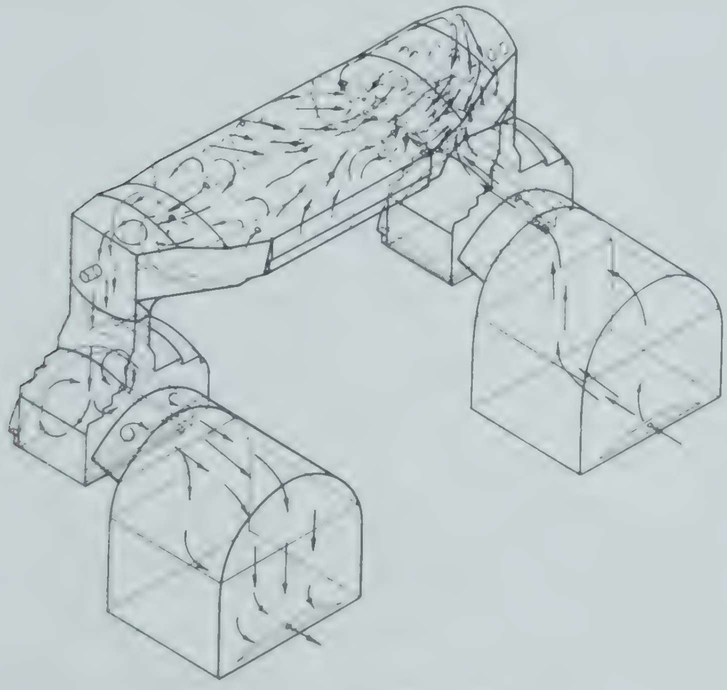


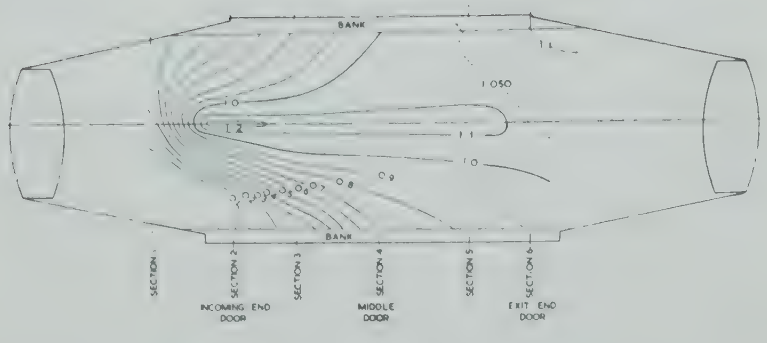
Fig. 3.—*General Flow Pattern*

Fig. 1 is a composite photograph which shows the flow pattern of the air in a furnace air-uptake. The air enters from the left at the bottom and then sweeps across to the right of the uptake. The result is that air flow is unevenly distributed across the uptake which, in turn, leads to an asymmetric distribution of combustion air in the furnace chamber. When using a water model, flow pattern features of this sort are immediately apparent. Although water models are usually less well suited to quantitative work, it is the rapidity of observation that makes them so useful, particularly if used in conjunction with an air model.

Use of Air Models

The disadvantage due to the difficulty of good visualisation in the air model is easily outweighed by the ease with which quantitative work is executed. This work may take the form of detailed traverses of air temperature, velocity, or gas concentration. In order to establish flow patterns or mixing rates in air models, the nozzle fluid may be heated or have injected into it a suitable tracer gas such as carbon dioxide,[6] helium, or nitrous oxide. The proportion of nozzle fluid to " combustion air ", the air : fuel ratio, may then be determined by measuring air temperature or sampling and analysing at points through the furnace system. The method used at Swinden Laboratories to label the nozzle fluid is to heat the air entering the model burner to a temperature of 200 to 300° C. by passing it through an electric heater. Air temperature in the model is then measured with 36 S.W.G. base-metal thermocouples connected to a potentiometric recorder.

Typical results obtained by this method are shown in Figs. 4 and 5. These mixing patterns were plotted during an investigation of open-hearth furnace roof wear.[5] Often the refractory roof of an open-hearth furnace may have to be rebuilt as a result of local wear in the vicinity of the back wall, although the rest of the roof is still serviceable. It was suggested that there may be a connection between this asymmetric distribution of wear and the asymmetry of flow pattern shown in Fig. 4. The flow pattern leads to fuel-rich mixtures reaching the roof near the back wall; the deleterious effects on refractories of a reducing atmosphere are well known. An investigation in the full-scale furnace established that there were fuel-rich regions under the roof at the back wall. Experiments were then performed on the model



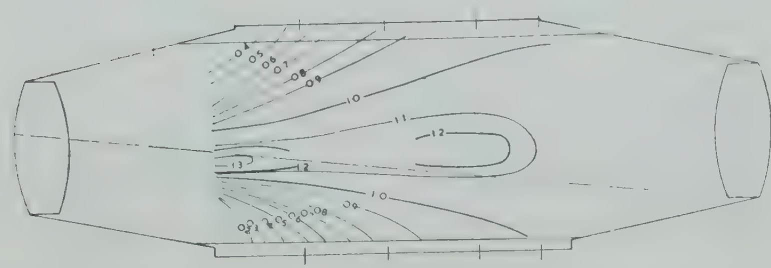


Fig. 4 (Top)—*Mixing contours at bath level with the burner in the normal position, in an air model of a single uptake open-hearth furnace.*

Fig. 5 (Bottom)—*Mixing contours at bath level with the burner turned 6° towards the front wall, in an air model of a single uptake open-hearth furnace.*

to determine the amount of burner movement necessary to eliminate the asymmetrical mixture distribution. The effects both of angular movement and parallel transposition were investigated. The effects of an angular movement may be judged from a comparison of Figs. 4 and 5.

The burners in a number of open-hearth furnaces were then moved. The results obtained showed that it was possible to make the flow pattern symmetrical and to make the roof wear more uniform.

A further case of a detailed investigation[5] of part of a furnace structure arose from the very high rates of wear of the roof of a 300 ton tilting furnace. The wear was local, taking place over a few square feet, occasioning delays whilst repairs were made.

Measurements of the rate of iron oxide deposition[18] on the roof showed that areas of heavy deposition were also areas of severe wear. Model work was then begun, the first object being to devise a technique which would reproduce the wear pattern obtained in the furnace. The technique used initially was similar to that described by Leys and Leigh.[19] In this method balsa-wood dust is introduced into the model to represent droplets of iron oxide. The roof of the model is coated with a suitable adhesive so that dust arriving at the roof of the model is retained. It was not found possible, during the initial tests, to make the dust deposition pattern coincide with the known wear pattern. This does not necessarily mean that it is not possible to obtain the right results using dust.

Alternative methods were then considered. The technique evolved is one in which sulphur-dioxide is injected into the model at the surface which represents the slag surface in the furnace after the charge is completely molten. With this method it is possible to produce a pattern which coincides with the pattern of roof wear.

The principle of the method is to allow the sulphur dioxide to enter the model at a controlled rate and to detect its first arrival at the roof by the coloration of filter paper, soaked in starch/potassium iodate solution, stuck to the underside of the roof. The relative rates of arrival of sulphur dioxide at different parts of the roof are determined by repeating the test but using a solution of starch, potassium iodate, and sodium thiosulphate. This solution gives a coloration only when all the hypo has been neutralised by the liberated iodine. The time between the appearance of colour when using the first solution and the appearance of colour at any point when using the second solution is inversely proportional to the rate of arrival of sulphur dioxide at that point.

Adequate similarity conditions being reached empirically a series of modifications were then tried on the model. These included alterations to the internal lines of the model, and the use of various types of protective air curtains.[21] Of the methods that were not ruled out by the severe practical limitations found at the furnace, that which seemed most promising was the use of a low velocity stream of air directed under the furnace roof in the region of heaviest wear. When tried in the furnace, this method did reduce the rates of deposit of fluxes on the roof and improved the roof life. During the three trials on the furnace the usual mid-life roof repair was not required.

Soaking Pits

A considerable use is made of models in a large variety of problems other than those connected with open-hearth furnaces.[22]

Work has been done on a 1/7th scale model of a soaking pit to investigate the effects of a proposed alteration to the outlet flue, to reduce the trouble caused by slag entering the waste gas system. The model work, using Reynolds similarity, indicated that the general flow pattern in the pit would not be affected by the alteration. It was noticed, however, that considerable short-circuiting of the combustion gases occurred, as shown in Fig. 2. Modifications were made to the model until conditions were obtained which resulted in a major flow forwards from the burner to the end of the pit, the gases then turning downwards and flowing towards the waste gas offtake. When the full-scale pit was modified similarly, it led to an improvement in temperature distribution in the pit, trouble having been encountered previously with overheated ingots near the burner.

Premelting of Scrap

Much work, including work on water models, pilot scale, and full-scale plant, has been done in an attempt to achieve a satisfactory method of premelting scrap using oil fuel.[23] The scheme was such that it was necessary to melt steel scrap in the same furnace body in which it would be refined later using the normal electric furnace arcs.

The method now being considered is that of counterflow melting in some sort of shaft furnace. This method has recently been suggested by Thring,[24] although several attempts have been made in the past,[25] particularly in Germany, to operate cupolas on a counterflow system.

Models are being used in this connection to assist in the determination of the factors which are of importance in the design of the furnace shape. Small-scale Perspex models are used with broken wax to represent the furnace charge. Hot air is used to melt the charge and to represent the combustion gases. Although wax is very convenient experimentally, it has the disadvantage that its latent heat of melting is about four times the heat quantity required to raise it to its melting point. In contrast to this, the latent heat of melting of iron is about $\frac{1}{4}$ of the heat quantity to raise iron to its melting point. For this reason the melting zone in the model tends to spread disproportionately.

Another important unknown factor is the magnitude of the adhesive forces which tend to hold a stack of heated and melting scrap together, so hampering free flow down the furnace. Even if these forces were known, it is not certain that a suitable material would be available to ensure similarity of behaviour. On the other hand, if factors such as distribution of gas flow are considered, the models, if run at the correct Reynolds number, may be relied upon to give the answer.

Conclusion

Furnace models have now been used on a wide variety of problems and have been of considerable value in enabling improvements to be made to full-scale plant. Experience indicates the adequacy of the similarity criteria used and gives confidence in the use of models when considering the adoption of relatively major changes in furnace lines or investigating the possibilities of new types of furnaces. Even in the wax melting experiments where the, as yet, unknown factors defeat any attempt at simulating certain conditions, the work has already been of value in stimulating new ideas and rejecting really bad ones.

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Symbols Used

A = area (sq. ft.).
G = burner thrust (lb. wt.).
g = conversion factor, gravitational to absolute units (32·2 ft./sq.sec.).
J = mechanical equivalent of heat (ft. lb./C.H.U.).
L = characteristic length (ft.).
M = mass flow rate (lb./sec.).
N_R = Reynolds number.
P = pressure (lb. wt./sq. ft. abs.).
R = gas constant (for air = 96·05 ft. lb./lb. ° C.).
$S = N_{Rm}/N_{Rp}$.
T = temperature (° K.).
t_n = nozzle temp. after adiabatic expansion (° K.).
V = velocity (ft./sec.).
γ = ratio of the specific heats C_p/C_v; (for air = 1·40).
η_n = nozzle efficiency.
μ = viscosity—absolute (lb./ft. sec.).
ρ = density (lb./cu. ft.).

Subscripts

$_a$ refers to ambient conditions.
$_e$ refers to equal density system.

$_f$ refers to density conditions at flame temperature.

$_i$ refers to inlet conditions before the nozzle.

$_m$ refers to model.

$_n$ refers to conditions in the nozzle throat.

$_p$ refers to prototype.

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THE DEVELOPMENT OF A NEW PROCESS (AUTOFINING) FOR THE CATALYTIC DESULPHURISATION OF PETROLEUM DISTILLATES

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SUMMARY

This paper describes the development of a process for the catalytic desulphurisation of petroleum distillates from the initial laboratory studies to the final design and construction of a large-scale unit. The intermediate step included operations on a pilot plant scale of between 100 to 300 barrels per day capacity. Details are given of the problems met with in the pilot plant work and the steps taken to overcome them in the final design of a 3500 barrels per day unit. The main problem was connected with the selection of the correct materials of construction in view of the highly corrosive nature of the recycle gases in the process.

Introduction

In the petroleum industry, a number of products require a reduction in their sulphur content, either for odour improvement or to make them less corrosive. The British Petroleum Company has been engaged for some years on research to develop a process which would desulphurise adequately both high boiling (kerosines and diesel oils) and low boiling distillates. The application of known processes was restricted either by feedstock limitations or high capital and operating costs. The outcome of this research was the development of a new process termed " autofining ". Details of the process with emphasis on the process variables have been given in earlier papers.[1, 2, 3]

The present paper is mainly concerned with the development of the process from its original conception to the final commissioning of large-scale units with emphasis on the scaling-up problems encountered both in the process and design.

Description of the Process

The autofining process, as finally developed, is a process for the catalytic desulphurisation of petroleum distillates at moderate pressures and temperatures, in which the sulphur compounds are preferentially attacked and converted into non-substituted molecules by the action of hydrogen, and in which the hydrogen required is provided by dehydrogenation of part of the feedstock itself. The sulphur is removed as hydrogen sulphide, which can easily be separated from the other reactants.

The gases, which are mainly hydrogen and are continuously recycled, join the cold feed before the heater as shown in Fig. 1. After vaporisation and super-heating to the required operating temperature, the vapours pass downwards through

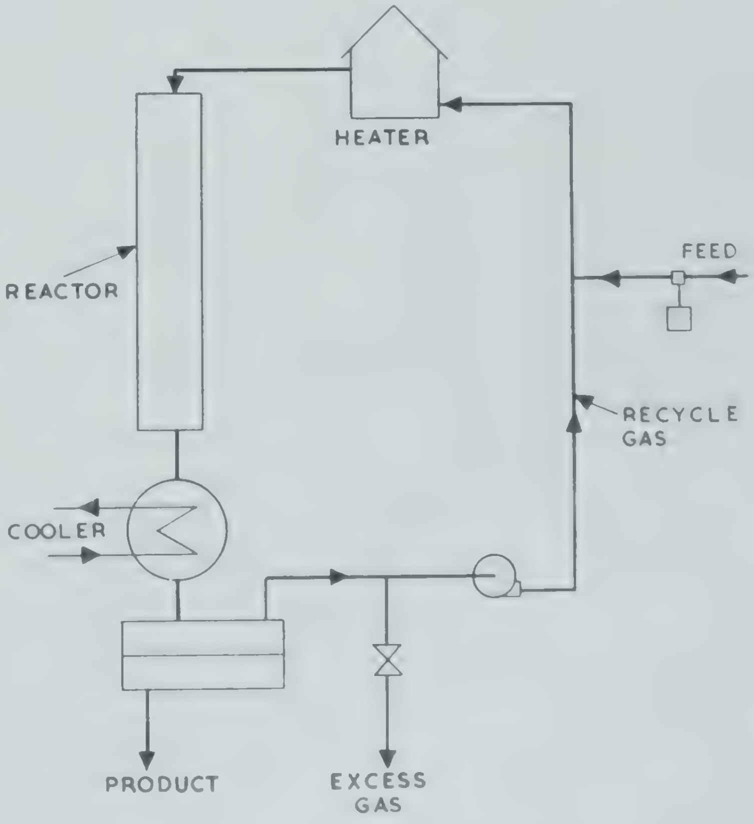

Fig. 1.—*The autofining process. Flow diagram*

the catalyst bed where the reactions take place. Under the preferred conditions, the reactants are in total vapour phase in the reactor. After cooling, the separated gases are recycled, and the liquid product is stabilised for the removal of dissolved gases and hydrogen sulphide. Control of pressure is maintained by venting the excess gas before recycling. It is not essential to remove hydrogen sulphide from the recycle gases, and this gas is allowed to reach equilibrium with the cooled liquid in the high-pressure separator so that all sulphur removed from the feedstock is obtained as hydrogen sulphide dissolved in the liquid product at plant pressure. During processing a little carbon is deposited on the catalyst, which ultimately leads to a loss of catalytic activity. The catalyst can be regenerated *in situ* with steam-air or inert gas-air mixtures to restore fully the original activity.

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The two main reactions in the process are dehydrogenation and desulphurisation which take place simultaneously and are typified by the following equations—

Dehydrogenation

$$\underset{\text{(methylcyclohexane)}}{C_7H_{14}} \longrightarrow \underset{\text{(toluene)}}{C_7H_8} + 3H_2 \qquad (1)$$

Desulphurisation

$$RSH + H_2 \longrightarrow RH + H_2S \qquad (2a)$$

$$R_1SR_2 + 2H_2 \longrightarrow R_1H + R_2H + H_2S \qquad (2b)$$

Reaction (1) refers to a naphtha feedstock but similar reactions are known to take place with the higher boiling feedstocks although the molecules are more complicated. Reaction (1) is controlled to give only sufficient hydrogen to satisfy reaction (2) and to provide adequate control of the process. There is therefore a slight increase in the aromatic content after autofining.

Reaction (2) results in a slight reduction in specific gravity due to removal of sulphur and also to some lowering of the boiling range. Material boiling below the initial boiling point of the feedstock may be formed; this depends upon the molecular weight of the sulphur compound and the position of the sulphur atoms in the molecule.

Reaction (1) is endothermic and reaction (2) exothermic and from a knowledge of the compounds in the naphtha boiling range (up to C_8) it has been shown that even if 1% weight of sulphur is removed and the reaction is in hydrogen balance, the heat consumed is only equivalent to a temperature drop of 3° F. of the reacting stream. Thus the autofining process can be considered as substantially thermo-neutral.

A catalyst of mixed cobalt and molybdenum oxides on alumina is preferred. Processing periods depend upon the boiling range of the feedstock and the space velocities employed; generally, with the lighter feedstocks, such as straight-run naphthas and kerosines, regeneration is required only at intervals in excess of 1000 hours, but with heavier feedstocks, such as diesel oils, the intervals are reduced to 200 to 400 hours.

Product recovery from autofining is very high; the major loss is that due to the removal of sulphur. Any losses due to the small gas-make and deposition of carbon on the catalyst are usually less than 0·1% by weight.

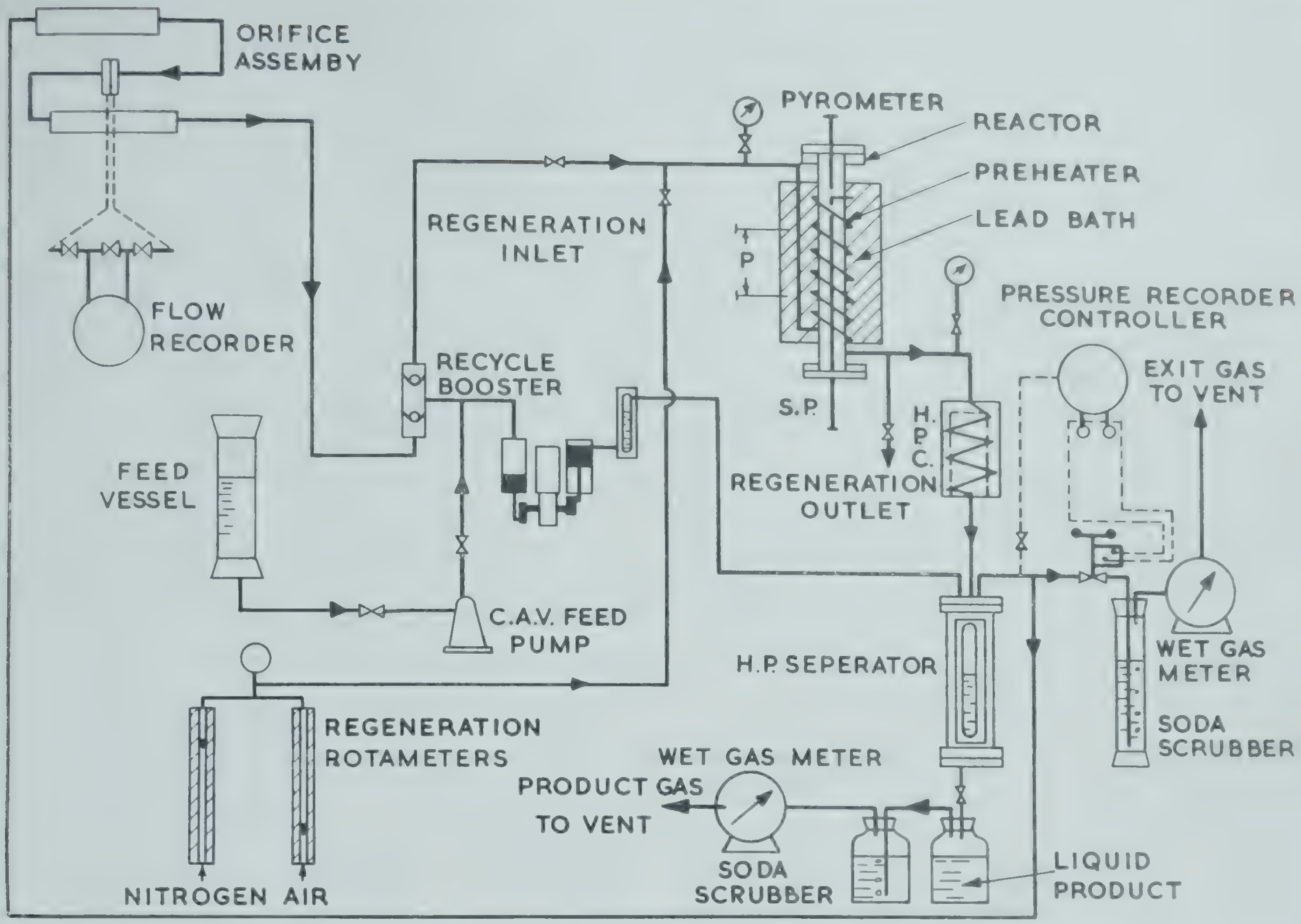
Laboratory and Small-scale Experiments

When the original idea had been proved by a few simple experiments, an intensive programme was started on laboratory and small-scale equipment to determine the optimum process conditions to be applied to a wide range of feedstocks. At this early stage, only laboratory samples of catalyst were available so that at the same time as the work on the process variables was proceeding, investigations were also pursued in conjunction with the manufacturers to prepare, by commercial methods, samples of catalyst which were subsequently tested on the small scale. A line diagram of the small-scale units employed in this work is given in Fig. 2. The essential parts of the equipment, *e.g.* the reactors and piping, were fabricated from 18/8 stabilised steel and the reactors had a catalyst capacity of 500 ml. and 1 litre. The units were isothermal.

One of the main difficulties in small-scale operations of this nature is to recycle the gases at constant rate without disproportionate losses. The general level of excess gas-make is low at the small throughputs employed in laboratory work, and even a small loss cannot be tolerated. A small gas booster was therefore designed, utilising the principle of the displacement-type plunger pump with a liquid seal on the glands[4] and this proved adequate for the recycling of very small quantities of gases without loss.

The process variables studied on these units included temperature, pressure, space velocity or feed rate, gas recycle rate, hours on stream, feedstock, and catalyst.

From the work on the process variables, it was possible to lay down the following set of process


ORIFICE ASSEMBY
PYROMETER
REACTOR
PREHEATER
LEAD BATH
REGENERATION INLET
P
FLOW RECORDER
PRESSURE RECORDER CONTROLLER
RECYCLE BOOSTER
EXIT GAS TO VENT
S.P.
H. P. C.
FEED VESSEL
REGENERATION OUTLET
C.A.V. FEED PUMP
WET GAS METER
H.P. SEPERATOR
SODA SCRUBBER
REGENERATION ROTAMETERS
WET GAS METER
PRODUCT GAS TO VENT
SODA SCRUBBER
LIQUID PRODUCT
NITROGEN AIR

P = PYROMETER
S.P. = SEARCH PYROMETER
H.P.C. = H.P. COOLER

Fig. 2.—*Small-scale catalytic plant*

conditions under which the majority of feedstocks could be economically desulphurised :—

Pressure	50 to 200 lb./sq. in. g.
Temperature	750° to 800° F.
Space velocity	1·0 to 5·0 v./v./hr. (volumes of oil/volume of catalyst/hour).
Recycle rate	2000 to 4000 cu. ft./bbl.
Catalyst	Mixed cobalt and molybdenum oxide on alumina.

Once the chemical form of the catalyst had been established, it became necessary to decide on suitable physical characteristics for commercial operation. The desirable physical properties were a high resistance to attrition, a high crushing strength, and suitable particle shape and size.

Working in conjunction with the catalyst manufacturers, a testing procedure and final specification was eventually drawn up which covered both granular and pelleted catalysts which could be further tested under commercial conditions in a larger pilot plant.

Pilot Plant Investigations

Following upon the successful outcome of the small-scale work, the decision was made to proceed with the investigations up to the design of a full-scale unit to be installed at the B.P. (Llandarcy) Refinery.

Investigations in the laboratory were necessarily limited and before a detailed design for a large-scale unit could be made, a number of points needed to be investigated on the pilot plant scale. It was decided therefore to erect a pilot plant at the B.P. (Grangemouth) Refinery to investigate the following points :—

(1) the effect on the process of translation to a large scale,

(2) the behaviour of the catalyst on prolonged use under commercial conditions, and

(3) the provision of operating and chemical engineering data for the design of a full-scale unit.

The B.P. (Grangemouth) Refinery was chosen as the site of the pilot plant because it already had available a number of ancillary items of equipment such as compressors, heaters, and heat exchangers which formed part of a disused catalytic polymerisation unit and only the reactor had to be specially designed.

As mentioned previously, the small-scale work was carried out in equipment fabricated substantially from stainless steel, a material which had been found to give excellent service. Small-scale experiments on the corrosion resistance of various steels carried out at the research station had shown that only steels of high chrome content were adequately resistant to hydrogen sulphide under the conditions expected in the autofining process. However, a much less costly unit could be constructed of mild steel, and the pilot plant at Grangemouth was therefore fabricated entirely of mild steel. The reactor was designed to be of sufficient strength to give a useful life even at the high corrosion rates expected.

Original design

The original design of the plant was based on a wide cut feedstock of 100–350° C. boiling range at a space velocity up to 5 v./v./hr. This, in conjunction with a catalyst volume of some 7 cu. ft., was equivalent to a throughput of 150 barrels per day. Before the plant was completed, the work at Sunbury showed that neat gas oil of about 200 to 360° C. boiling range could be autofined satisfactorily without the addition of a lighter material to provide the hydrogen for the conversion of the sulphur to hydrogen sulphide, and it was decided that the pilot plant should start up on this feedstock. This change meant a considerable reduction in throughput which seriously under-loaded some of the equipment. For example, the original reactor was designed for a maximum heat loss of 10 000 B.t.u./hr., corresponding to a temperature drop of 6° F. when the throughput is 150 barrels per day of wide cut feedstock. With a gas oil feedstock, where it was expected that space velocities as low as 1 v./v./hr. might be employed, the throughput would be down to 30 barrels per day and a temperature drop of 30° F. might result. As this drop was intolerable, a second reactor was designed which, although it was four times the size of the first, was more efficiently insulated and the heat loss was only slightly higher than of the smaller vessel. The larger reactor was erected alongside the original reactor so that either could be operated depending upon the feedstock and flow rates (v./v./hr.) required.

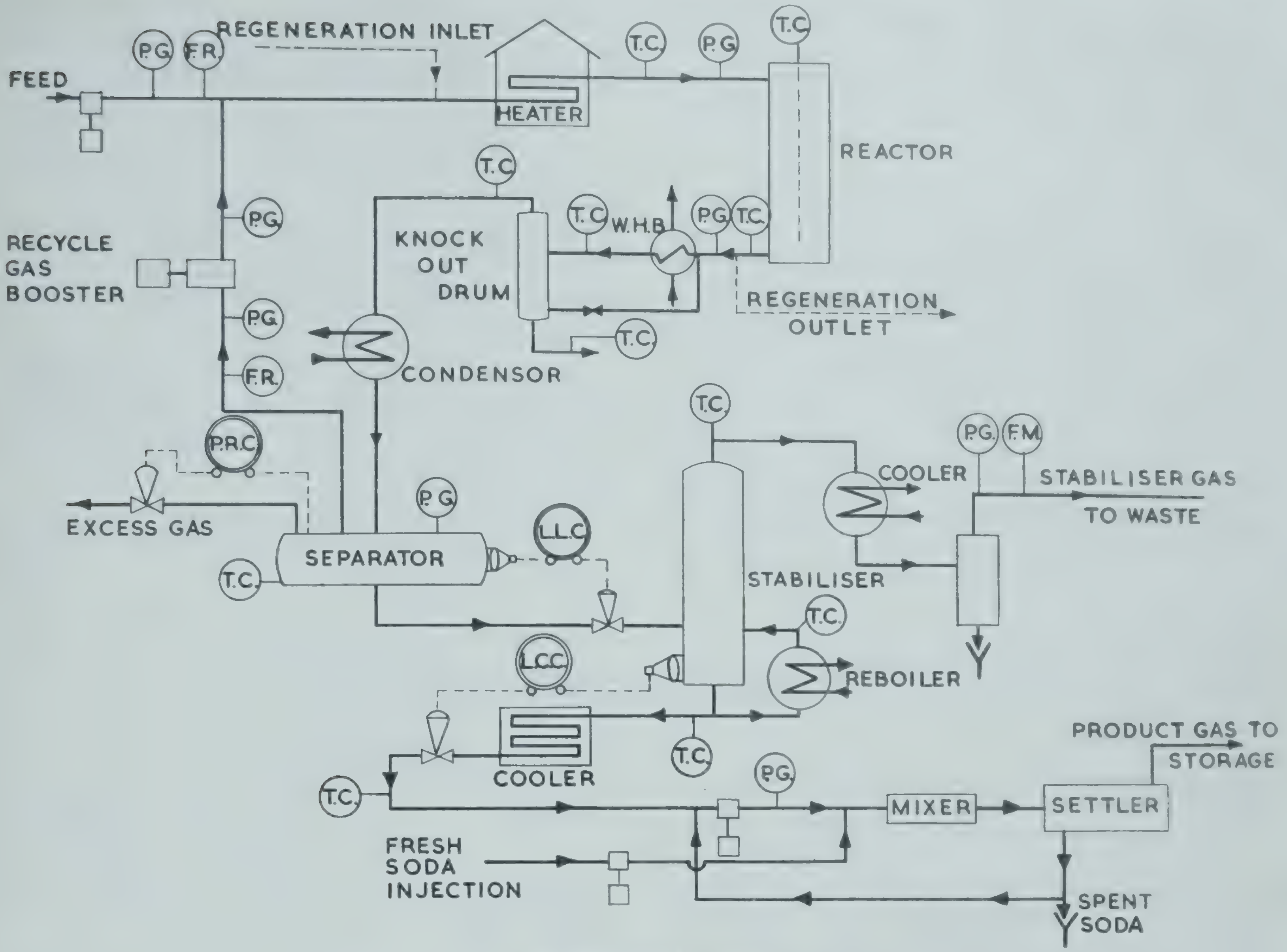
A simplified flow diagram of the unit is given in Fig. 3.

Original reactor

The original reactor was fabricated from a length of standard 12 in. inside diameter mild steel pipe. The top and bottom joint rings were made originally of aluminium alloy of elliptical section, fitting into recesses on the two flanges. These rings were found to be unsatisfactory because they softened at the higher temperatures used during regeneration. They were replaced by soft iron joints, which gave satisfactory service. The joints on the reactor inlet and outlet lines were of the same type.

Pipework throughout the high pressure part of the unit was of 2 in. inside diameter mild steel. On auxiliary equipment, such as the stabiliser, $1\frac{1}{2}$ in. inside diameter pipe was used and for lighter duties, such as excess recycle gas lines, $\frac{1}{2}$ in. and 1 in. inside diameter pipe was employed.

With certain feedstocks a separate redistillation step is necessary to give products of specified colour. To avoid this separate step, it was suggested that a knock-out tower could be incorporated at the reactor outlet to remove some 1–2% volume of heavy ends. For this tower to operate successfully it was necessary for it to be closely controlled at a temperature near the dew point of the reactants and further consideration suggested that a waste heat boiler would be more suitable for this purpose than a conventional heat exchanger. The necessary equipment was therefore incorporated in the pilot plant at a later date (see Fig. 3). Under the new arrangement, the combined stream of feed and recycle gas passed direct to the pipe-still and thence to the reactor, the heat exchanger having been replaced by the boiler. From the reactor outlet, product gases were passed through the boiler and into the tower, the temperature being controlled by varying the steam pressure in the boiler. Provision was also made for part of the reactor products to bypass the boiler and enter the tower direct through the bottom inlet only. This bypass stream did duty as a reboiler, supplying extra heat to the tower base as and when required and was measured by a flow recorder coupled to alternative orifice plates giving full-scale readings of 50% and 100% by volume of throughput. The reactor, tower, and boiler were placed as close together as the pipework fittings would allow, to reduce to a minimum the heat loss from the transfer lines. Overhead product from the tower


FEED
P.G.
F.R.
REGENERATION INLET
HEATER
T.C.
P.G.
T.C.
REACTOR
T.C.
KNOCK OUT DRUM
T.C.
W.H.B.
P.G.
T.C.
REGENERATION OUTLET
T.C.
RECYCLE GAS BOOSTER
P.G.
P.G.
F.R.
CONDENSOR
P.R.C.
EXCESS GAS
P.G.
SEPARATOR
L.L.C.
T.C.
T.C.
COOLER
P.G.
F.M.
STABILISER GAS TO WASTE
STABILISER
T.C.
REBOILER
L.C.C.
COOLER
T.C.
T.C.
P.G.
PRODUCT GAS TO STORAGE
MIXER
SETTLER
FRESH SODA INJECTION
SPENT SODA

W.H.B. = WASTE HEAT BOILER

T.C. = Thermocouple.
L.L.C. = Liquid level controller.
P.R.C. = Pressure recorder controller.
F.R. = Flow recorder.
F.M. = Flow meter.
P.G. = Pressure gauge.

Fig. 3.—*150 B.P.D. autofining pilot plant*

passed into the old product line and so to the product coolers, whence it was disposed of as before.

Results of pilot plant work

On the process side, the desulphurisation obtained and the quality of the products were in complete accord with the small-scale work and no scaling-up problems were apparent. In particular the catalyst, which had been removed on a number of occasions for various reasons over a period of two years, retained practically all its original activity after 34 regenerations. No indications of excessive pressure drop through the formation of fines were observed, although some variations in physical properties were noticeable, most of which can be attributed to mechanical breakdown when dropping the catalyst charge.

The main chemical engineering problems shown up by the pilot-plant work included :—

(*a*) very high corrosion rates of mild steel equipment such as transfer lines, reactor vessel, heat exchanger tubes, pipestill tubes, etc., were experienced when temperatures exceeded 500° F. At 800° F., corrosion rates as high as 0·15 in. per year were recorded. The corrosion was due to the hydrogen sulphide present in the recycle gas,

(*b*) carry-over of corrosion products from the pipestill and transfer lines into the reactor vessel occurred, the effect of which was to cement together the particles of catalyst in the top of the bed, and

(*c*) very low heat transfer co-efficients were achieved in the various items of heat exchanger equipment.

Design and Construction of a Large-scale Unit

To illustrate the problems arising in the design of a full-scale unit, the design of the 3500 B.P.D. unit engineered for Llandarcy will be discussed. A flow diagram of the unit showing the operating conditions is given in Fig. 4.

The plant had to be capable of treating three separate feedstocks, these being batched through the plant in turn at intervals to suit both the production demands for the individual products and the catalyst condition.

Sequence of operations

The process was visualised as being broken down into the following sequence of operations :—

(*a*) preparing a suitable mixture of feedstock and recycle gas at a temperature of 800° F. and a pressure of 100 lb./sq. in. g.,

(*b*) passing this mixture through a catalyst bed to generate hydrogen and convert the various sulphur compounds present in the feedstock to hydrogen sulphide,

(*c*) cooling the autofined mixture to atmospheric temperature to condense the autofined product and permit its separation from the recycle gas, the latter then being returned to the process by compressors,

(*d*) removing any hydrogen sulphide, hydro-

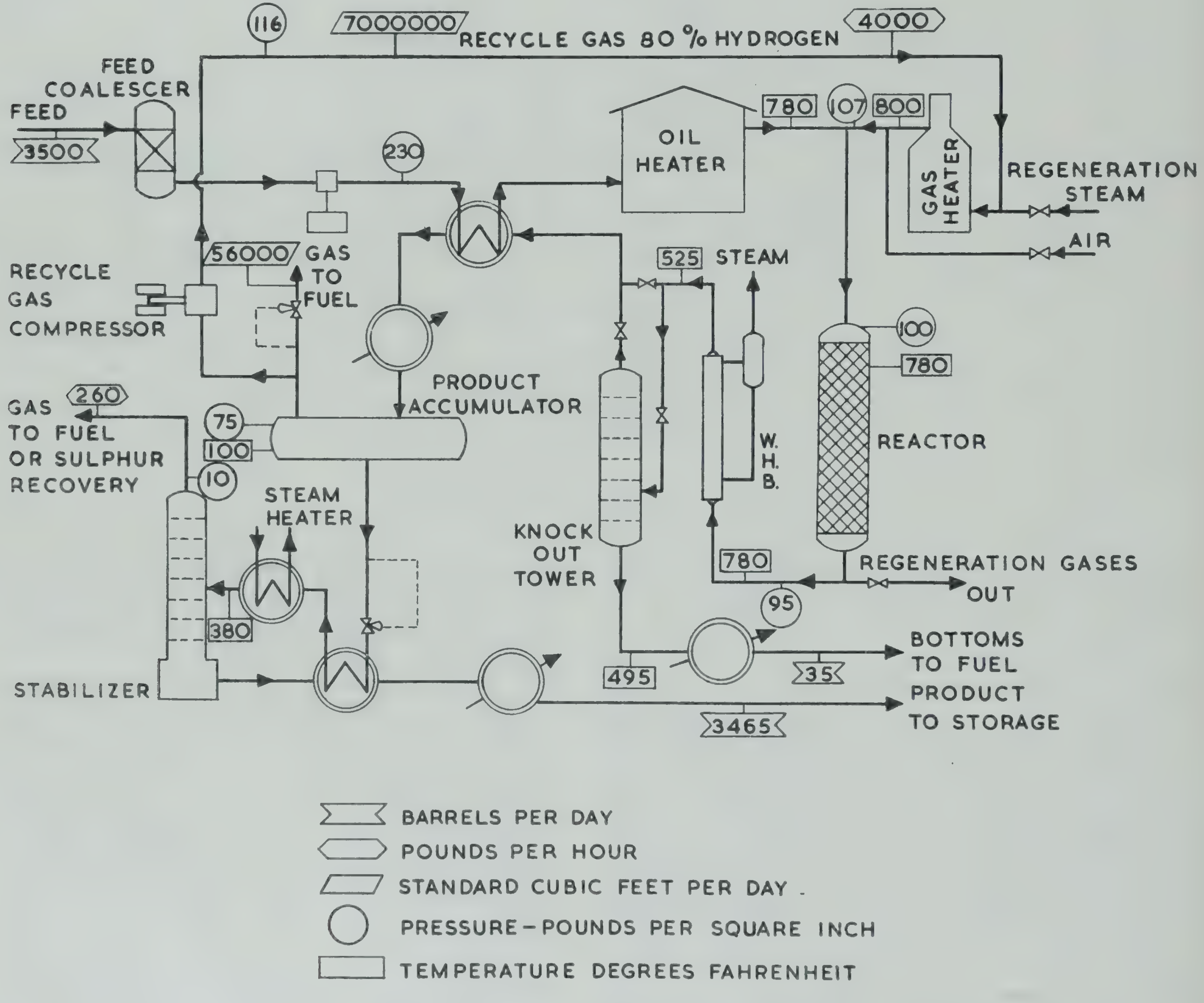

116
7000000
RECYCLE GAS 80 % HYDROGEN
4000
FEED COALESCER
FEED
3500
230
OIL HEATER
780
107
800
GAS HEATER
REGENERATION STEAM
AIR
56000
GAS TO FUEL
RECYCLE GAS COMPRESSOR
525
STEAM
100
780
PRODUCT ACCUMULATOR
GAS TO FUEL OR SULPHUR RECOVERY
260
75
100
10
W. H. B.
REACTOR
STEAM HEATER
KNOCK OUT TOWER
780
REGENERATION GASES OUT
95
380
BOTTOMS TO FUEL
STABILIZER
495
35
PRODUCT TO STORAGE
3465
BARRELS PER DAY
POUNDS PER HOUR
STANDARD CUBIC FEET PER DAY
PRESSURE – POUNDS PER SQUARE INCH
TEMPERATURE DEGREES FAHRENHEIT

Fig. 4.—*Flow sheet of 3500 B.P.D. autofining unit*

gen, methane, and other dissolved gases from the autofined product, and

(*e*) regenerating the catalyst at suitable intervals.

Corrosion

As already stated, one of the main problems was that of corrosion due to the hydrogen sulphide in the recycle gas. This not only affects the life of the plant but can also cause deposition of corrosion products in the top of the catalyst bed, which leads to excessively high pressure loss through the equipment and to a reduction in throughput.

Corrosion could be reduced, if not altogether stopped, by removing hydrogen sulphide from the recycle gas. The cost of the equipment required would have been rather high and the plant too small to have any financial advantage from the possible recovery of sulphur. In view of this, and because it was considered desirable to keep the first full-scale plant as simple as possible, it was decided not to remove hydrogen sulphide from the recycle gas, but to fabricate any equipment coming into contact with recycle gas at a temperature above 500° F. in corrosion resistant materials.

Experience with test pieces at Sunbury and Grangemouth showed that both 18/8 stainless steel and calorised steel are resistant to the conditions, but steels of the 4/6% chrome group were found to be less resistant than mild steel. It was decided to adopt 18/8 stainless steel. Calorised steel may have been somewhat cheaper but flaking of the coating at tube rolls in furnace headers, and pinhole imperfections, might give rise to severe localised corrosion.

Reactor

The first thing to determine was the number of reactors to be used. With the types of feedstocks to be handled in this plant, indications from pilot-plant experiments were that long periods on stream could be expected before the catalyst required regenerating. As a regeneration period of 24 hours about once a month would suffice, this made it possible to use only one reactor, the resulting simplification of plant arrangement more than off-setting the relatively small decrease in utilisation of the plant.

It had been decided that the gas should flow downwards through the catalyst bed. To give good gas distribution without excessive pressure drop, and also to give an economical size of vessel, a height to diameter ratio of approximately 3 to 1 was chosen for the bed. In the design of the reactor, the choice lay between a stainless steel vessel externally lagged, or a mild steel vessel, lined internally with a refractory insulating concrete "gunited" into position. Internal or external insulation is essential in order to reduce heat losses, a relatively small drop in temperature having an adverse effect on the autofining reactions. Internal insulation was thought to have the following advantages :—

(*a*) the shell of the vessel is kept well below the temperature at which there is a falling off in the allowable working stress of mild steel thereby enabling the thickness of the vessel walls to be kept to a minimum,

(*b*) it serves to shield the vessel walls from any contact with corrosive gases, and

(*c*) even in the case of a slight gas breakthrough to the shell of the vessel, the rate of corrosion should be very small due to the reduced temperature of the shell and the poor gas circulation.

It was decided to use a mild steel vessel, internally lined with refractory insulating concrete, the lining being 4 in. thick and retained in position by adhesion to the walls and by the use of wire jaggers. Horizontal rings welded at 2 ft. intervals around the inside of the vessel also helped to support the lining, but their main function was to prevent vapour from by-passing the catalyst bed. With this arrangement the temperature of the vessel shell was estimated to be 325° F.

To ensure good gas flow through the catalyst bed, attention was paid to the design of the gas distributing and collecting arrangements and these were fabricated in 18/8 stainless steel. The catalyst was supported on stoneware balls.

Preparation of oil/recycle gas mixture

The following methods were possible :—

(*a*) To preheat the required mixture of feedstock and recycle gas in a series of heat exchangers and bring to final temperature in a fired heater. This method has several disadvantages, namely : (1) separation of feedstock from the recycle gas mixture can occur in exchangers, (2) all the equipment is exposed to the corrosive action of the hydrogen sulphide in the recycle gas, and (3) horsepower requirements for the recycle gas compressors are at a maximum unless a multipass heater is used.

(b) To vaporise and superheat the oil in a series of exchangers and fired heater and then mix with cold recycle gas. From the aspects of corrosion and compressor loading such a method would have several advantages, but unfortunately the degree of super-heat of the feedstock to allow for mixing with cold gas causes too much thermal cracking.

(c) To vaporise the feedstock in exchangers and fired heater and then blend with recycle gas brought up to temperature in a separate heater.

Method (c) was adopted for the following reasons :—

(1) With the feedstocks to be used, corrosion, on the cold-oil side of the heat exchangers and in the oil heater, should be negligible when using ordinary mild steel equipment. The heat duty for raising the temperature of the oil to 800° F. is several times that required for raising the recycle gas to the same temperature. Thus the main heat load of the plant can be developed in ordinary mild steel equipment and the use of alloy material minimised.

(2) Only a relatively small fired heater would be required for heating the recycle gas, so that even if all its tubes and headers were fabricated in 18/8 stainless steel, the cost should not be high.

(3) The design heat load of the gas heater would make it very suitable for use as a steam superheater during the regeneration period.

(4) Since only a relatively small gas heater would be required, the pressure drop through it should be low and the horse power requirements of the recycle gas compressor kept down.

(5) The problem of oil separating from a mixture of oil and recycle gas on its way to a heater is avoided.

Since the heat duty of the recycle gas heater was small, a simple radiant furnace was adopted. It was decided to forego the slight increase in overall thermal efficiency which could have been obtained by installing a convection bank in this heater.

Since only the recycle gas heater would be required during the regeneration period, it was not possible to combine the coils of the recycle gas heater and the feedstock heater in one chamber.

Cooling the reactor products

The next step was to arrange for the cooling of the reactor products from 780° F. down to atmospheric temperature. To minimise the amount of alloy material required, rapid cooling to 500° F. was desirable. This called for equipment in which a good heat transfer rate could be obtained, preferably by a combination of high film coefficients and a high log mean temperature difference. A demand for high pressure steam at 235 lb./sq. in. g. arose in a later part of the process and for which refinery steam at 175 lb./sq. in. g. was unsuitable. A satisfactory solution to the rapid cooling to 500° F. and the production of high pressure steam was found in the use of a waste heat boiler. This was a vertical heat exchanger, single pass in shell and tubes, the reactor products passing through the tubes at high velocity. Water circulates through the shell, the resulting steam/water mixture being taken into a separate drum for separation. The tube sheets, tubes, and the channel at the inlet end of the exchanger are 18/8 stainless steel, the remaining parts and the drum are mild steel. High pressure steam not required by the autofiner passes into the refinery steam main.

Having been cooled to a temperature of 500° F. and thus rendered relatively non-corrosive, the reactor products are then passed through the shells of a series of mild steel heat exchangers and coolers. Cold, non-corrosive feedstock, on its way to the oil heater, passes through the tube side of the exchangers and water through the tubes of the coolers. By routing the various streams in this manner and using well-baffled shells, it was hoped to obtain good heat transfer coefficients. In order to avoid the hold up of liquid in the shells, the baffles are of the segmental, vertical cut type to give side to side flow and the exchangers and coolers mounted one above the other. The design of the heat exchangers and coolers was complicated to a certain extent because the plant had to deal with three separate feedstocks, each of which has its own heat load and temperature distribution throughout the plant.

Separation of recycle gas from autofined material

The reactor products, after being cooled to atmospheric temperature, are then separated in a simple horizontal vessel. The size of the vessel required for separation was not particularly large, but in view of certain factors such as the absorption of hydrogen by the catalyst after regeneration, the tailing off in hydrogen production as the process proceeds, the vessel was made much larger than would otherwise be required in order to provide reasonable gas storage capacity.

Removal of hydrogen sulphide from the autofined product

The liquid from the separator contains appreciable quantities of hydrogen sulphide together with other dissolved gases such as hydrogen and methane. To remove these, and at the same time make any slight adjustment to the flash point, the liquid from the separator is passed through a series of heat exchangers to a stripper column operating at 5 lb./sq. in. g. This arrangement is conventional; the product is removed from the column as a bottoms-make and, after cooling, is washed with caustic soda to remove any traces of hydrogen sulphide remaining before going to storage. The gases rich in hydrogen sulphide released from the reflux drum of this column are burnt in one of the heaters.

Regeneration

With the feedstocks envisaged it would be necessary to regenerate the catalyst once per month, the plant being taken off stream for a period of about 24 hours. A steam/air mixture at 800° F. would be passed over the catalyst, the steam being superheated in the heater normally used for the recycle gas. The gases from the reactor were to be vented to the stack.

Air/flue gas could have been utilised instead of air/steam but it was considered that the equipment required was not justified for dealing with the relatively infrequent regeneration.

To minimise the possibility of excessive temperatures during regeneration, which could adversely affect the catalyst, the reactor was equipped with thermocouples at a number of points.

Colour correction of tractor vaporising oils

To a certain extent the design of the full-scale plant was somewhat ahead of the full pilot plant programme and some little time after design had begun it was found that autofined tractor vaporising oil would not meet colour requirements although satisfactory in all other respects. To improve the colour would normally have meant re-distilling the tractor vaporising oil to remove 1–2% of the material as a bottoms product.

This would have necessitated an expensive distillation unit and would have considerably increased the operating costs, so other means were sought. The final solution was to insert a knock-out column in the heat exchanger string downstream of the waste heat boiler in the autofining circuit. In this column sufficient heavy boiling point bodies will be removed from the reactor products stream by carefully controlled partial condensation before the main condensation is allowed to take place.

Reduction of hydrogen losses

For the process to proceed satisfactorily, hydrogen losses must be minimised. Special attention was paid to the compressor glands and screwed pipework fittings were avoided where possible. If this could not be done, such fittings were seal welded.

Instrumentation

No special problems arose in connection with the instrumentation of the plant. The main streams, including steam and air for regeneration, were flow-controlled and the oil vapour and recycle gas streams temperature controlled at the heater outlets. Thermocouple points at 2 ft. intervals down the depth of the reactor were installed in order to ensure adequate temperature control of the catalyst bed during the regeneration period, it being essential to prevent the development of excessive temperatures which would injure the catalyst.

As the amount of bottoms to be removed from the knock-out column was rather small and the dew-point somewhat variable, according to the pressure and composition of the gas stream, the amount of reflux required was not regulated by the temperature of the gases leaving the top of the column. Instead, bottoms were drawn off from the column at some predetermined rate by a flow controller, a level controller in the base of the column controlling the amount of reflux required to bring about the necessary degree of condensation to maintain constant level conditions in the base of the column.

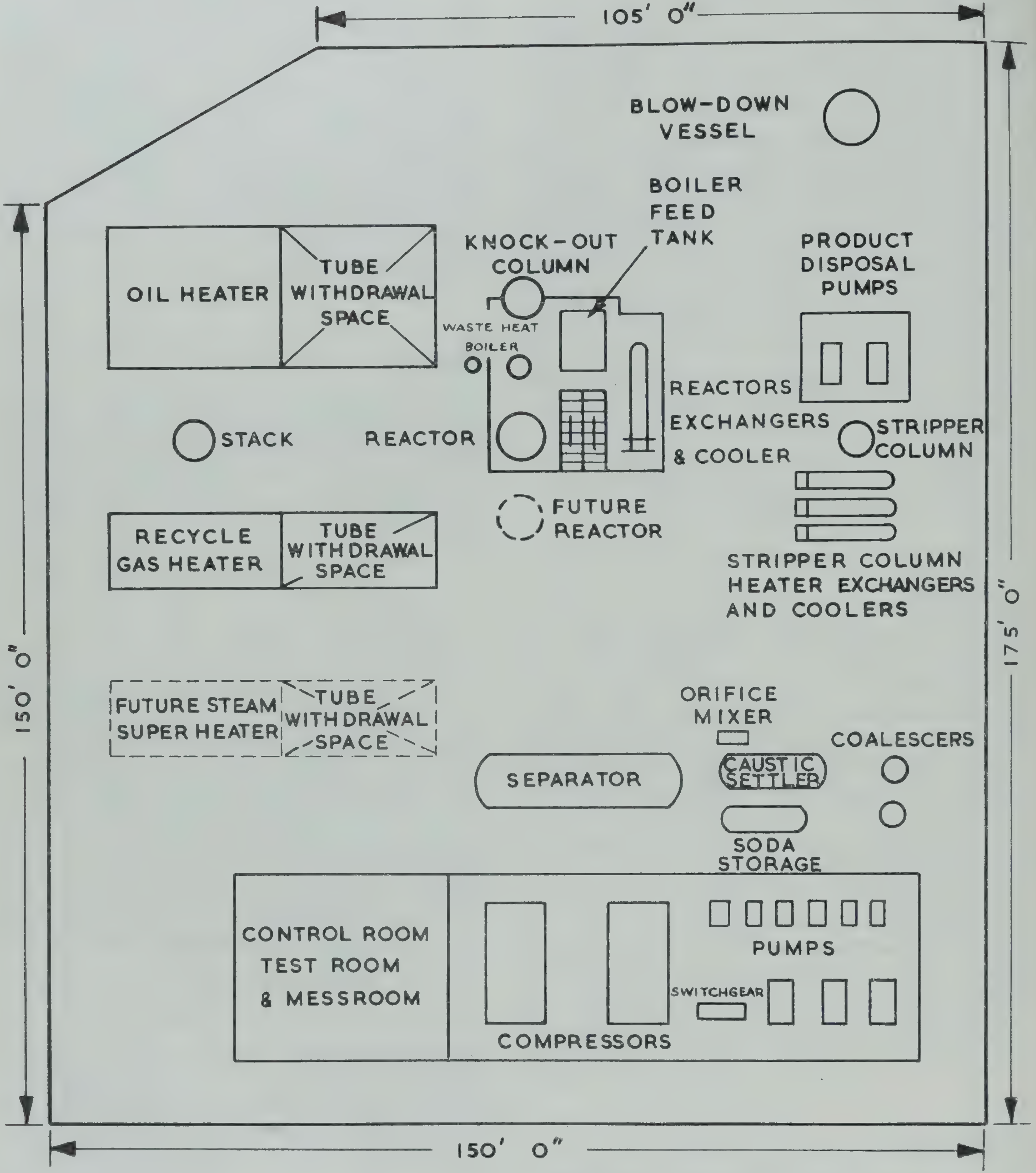

105' 0"
BLOW-DOWN VESSEL
BOILER FEED TANK
KNOCK-OUT COLUMN
PRODUCT DISPOSAL PUMPS
OIL HEATER
TUBE WITHDRAWAL SPACE
WASTE HEAT BOILER
REACTORS EXCHANGERS & COOLER
STACK
REACTOR
STRIPPER COLUMN
FUTURE REACTOR
RECYCLE GAS HEATER
TUBE WITHDRAWAL SPACE
STRIPPER COLUMN HEATER EXCHANGERS AND COOLERS
175' 0"
150' 0"
FUTURE STEAM SUPER HEATER
TUBE WITHDRAWAL SPACE
ORIFICE MIXER
COALESCERS
SEPARATOR
CAUSTIC SETTLER
SODA STORAGE
CONTROL ROOM TEST ROOM & MESSROOM
PUMPS
SWITCHGEAR
COMPRESSORS
150' 0"

Fig. 5.—*Plant layout of final unit*

TABLE I.—*Stages in Development of the Process*

Plant	Laboratory units	Pilot plant	Large-scale unit
Throughput at 3·0 v./v./hr.	0·5 B.P.D.	(*a*) 90 B.P.D. (*b*) *ca.* 300 B.P.D.	3500 B.P.D.
Reactor dimension	3 ft. × $1\frac{1}{8}$ in.	(*a*) 10 ft. 9 in. × 12 in. (*b*) 12 ft. 6 in. × 1 ft. 9 in.	23 ft. × 5 ft. 9 in.
Catalyst volume	0·018 cu. ft.	(*a*) 7·4 cu. ft. (*b*) 24 cu. ft.	288 cu. ft.
Materials of construction	18/8 chrome nickel stainless steel	Mild steel	Mild steel with refractory lining.
Insulation	Isothermal, heater windings and external lagging	$2\frac{3}{4}$ in. thick high temperature heat resisting plastic and $2\frac{1}{2}$ in. of 85% magnesia plastic applied externally and contained in a light steel container	4 in. refractory lining inside the reactor shell.

Layout

A flow sheet having been developed and the types and sizes of equipment together with the materials to be used for construction having been determined, a layout was then required. In preparing the layout, the following factors were borne in mind :—

(*a*) All lines containing recycle gas were kept as short as possible in order to keep down the compressor load.

(*b*) To reduce the temperature drop in the transfer line to a minimum, the reactor was positioned as close as was safe to the fired heaters.

(*c*) The separator and the various heat exchangers and coolers handling the reactor products were arranged at suitable relative levels to ensure adequate drainage of their shells.

(*d*) The equipment was split up into suitable groups, separated from one another by lanes which not only provide access, but also act as fire barriers.

To aid the development of a suitable layout, a model was constructed. Of the many layouts investigated, the one adopted is shown in plan in Fig. 5. From this, it will be seen that the above-mentioned requirements have been met. Space has also been provided for a second reactor if desired at some future date. If a second reactor is ever installed and the plant operated with one reactor on stream whilst the other is being regenerated, a separate steam superheater will be required. Provision has therefore been made in the layout for this.

In addition to the Llandarcy unit, which was commissioned in July 1952, the company has another unit of the same size at Aden refinery and the Grangemouth Pilot Plant has also been enlarged to a 500 B.P.D. commercial unit. The time between the beginning of the initial investigations and the commissioning of the first large-scale unit was approximately five years.

Acknowledgment

The authors wish to express their thanks to the Chairman and Directors of the British Petroleum Company Limited for permission to publish this paper.

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SCALE-UP METHODS FOR CONTINUOUS FILTRATION EQUIPMENT

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SUMMARY

In any continuous filter application, two or more of the following rates are involved:

(1) filter cake formation rate,
(2) filter cake dewatering rate,
(3) filter cake washing rate, or
(4) filter cake thermal drying rate.

The slowest rate will generally determine the size of the full-scale equipment, but for accurate scale-up, all applicable rates must be investigated.
In this paper a correlating method is developed for each rate by a theoretical approach but supported by actual plant later. Examples are included to show how the correlating methods may be used to scale-up filtration data so as to obtain a plant of maximum economic size.

Introduction

The aim of this paper is to describe scale-up methods which are of general application to continuous filtration equipment. Although occasional references to the details of particular types of filter are essential, it is not intended to describe the many different kinds of filter ; this subject has already been well treated in the literature.[1, 4, 5, 6, 9, 12, 13, 15, 16, 17]

To understand the approach, it must be realized that all continuous vacuum or pressure filters depend upon the same basic principle of a cyclic process composed of at the most five stages:

(1) cake is formed,
(2) cake is dewatered to a particular moisture content,
(3) cake is washed to remove valuable or undesirable solubles,
(4) cake is dried thermally, and
(5) cake is discharged.

Although the method for discharging a particular cake must be carefully established, it is clearly not normally an operation which will influence the scale-up of equipment. On the other hand, the rate at which one or more of the other operations can be carried out will critically affect the size of filter required. Where more than one of these rates is critical, great care must be taken to give due attention to each factor. It is therefore proposed to consider each of these four rates separately, showing how a suitable synthesis may be achieved where necessary. In doing this, attention will be drawn to the restrictions which the geometry of a filter automatically imposes on the way the operating cycle can be divided between the five stages.

Filtration data may be obtained either on laboratory scale, using what is known as the " leaf-test procdeure "[5, 9, 10, 18, 21, 22] or on pilot plant. Even if pilot-plant work is intended, it is always advisable to carry out a preliminary leaf-test investigation since the flexibility of the method enables the many variables to be examined rapidly. For this reason, the following discussion of how data may be correlated and extrapolated generally refers to leaf-test investigations but the same principles are naturally applicable to pilot-plant work.

It must be emphasised that attention is here devoted to data obtained from a slurry in its existing condition. It frequently occurs that the filtration characteristics of a slurry may be improved by such means as pH adjustment, the addition of flocculating or wetting agents, or preliminary thickening. While such methods of pretreatment are often very useful, it is not intended to discuss them in this paper.

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Scale-up from Cake Formation Rate

Theoretical background

Although more rigorous equations have been developed, the well-known modification of the Poiseuille equation[3] enables the important variables to be correlated simply :

$$\frac{dV}{Ad\theta} = \frac{\Delta p}{\mu\left(\frac{\alpha W V}{A} + r\right)} \qquad . \qquad . \qquad (1)$$

Equation (1) gives the instantaneous filtration rate after a volume, V, of filtrate has passed through the filter. To apply it to the cyclic process of continuous filtration, the equation must be integrated. Before doing this, the resistance terms will be discussed briefly.

With proper hydraulic design the resistance of the internal piping and drainage system can be made negligible and will be so assumed in this paper. Following common practice, it will also be assumed that the resistance of the filter medium is negligible although it is realised that this is not always true, either where the medium is very tight or where it is partly blinded. It will be seen below, however, that this assumption can often be justified by actual data.

The specific cake resistance, α, is unfortunately not constant but is influenced by many factors, the most important being :

(1) particle size distribution,
(2) particle shape characteristics,
(3) cake homogeneity,
(4) solids orientation in the filter cake,
(5) feed solids concentration, and
(6) pressure drop.

For a particular slurry, provided proper care is taken to form a homogeneous cake, the first five factors can generally be regarded as constants ; failing this, they must be handled as parameters since no satisfactory theoretical relationship has been established. The influence of pressure drop, however, has been expressed mathematically in the form of equation (2) :

$$\alpha = \alpha'(\Delta p)^{s} \qquad . \qquad . \qquad (2)$$

From the above discussion, equation (1) is restricted by certain parameters and, after simplification, can be integrated between the limits $\theta = 0$ and θ_f and $V = 0$ and V_f (where V_f is the volume of filtrate obtained in cake-forming time θ_f).

$$V_f/A = \left[\frac{2(\Delta p)^{(1-s)}\ \theta_f}{\mu\alpha' w}\right]^{\frac{1}{2}} \qquad . \qquad (3)$$

Equation (3) expresses filtration rate in terms of one cycle or revolution of the filter, whereas it is more convenient to utilise a time basis. This can easily be done by dividing through by the cycle time, θ_c ; since $\theta_f = B \times \theta_c$ where B is the fraction of the cycle devoted to cake forming, we obtain

$$\frac{V_f}{A\theta_c} = Y = \left[\frac{2(\Delta p)^{1-s}\, B}{\mu\alpha' w\theta_c}\right]^{\frac{1}{2}} \qquad . \qquad (4)$$

where Y is the filtration rate expressed in volume of filtrate per unit area per unit time. By multiplying this by a conversion constant, the filtration rate may be expressed in weight of cake per unit area per unit time.

Equation (4) shows how the variables would be expected to affect filtration rate.

Cake formation time

The cake formation time $\theta_f = B \times \theta_c$. Thence θ_f may be altered either by varying B or by varying θ_c. Equation (4) shows that at constant B, filtration rate is inversely proportional to the square root of either θ_c or θ_f ; therefore, a plot on logarithmic coordinates of Y as a function of θ_c or θ_f should give a straight line of slope $-0{\cdot}5$. Similarly, at constant cycle time (θ_c), the filtration rate is directly proportional to $\sqrt{B}$ and a similar plot should therefore give a straight line of slope $+ 0{\cdot}5$.

In practice, straight lines are usually obtained, thus showing the validity of the assumption concerning the filter medium resistance term, r, in equation (1). The slopes of the lines frequently deviate from $\pm 0{\cdot}5$, however, due to the specific cake resistance α altering during the filtering cycle, probably because of the migration of fines within the cake.

For example, in an investigation of the filterability of melamine crystals in aqueous ammonia, a logarithmic plot of dry solids/hr. sq. ft. as a function of cake-formation-time gave a straight line of slope $- 0{\cdot}617$. Examination showed that the equation for the line was

$$Y = 56{\cdot}0\,(\theta_f)^{-0{\cdot}617} = 56{\cdot}0\,(\theta_c B)^{-0{\cdot}617} \qquad . \qquad (5)$$

In this instance, where a bottom-fed vacuum drum filter was used, $B = 0{\cdot}25$ since the need for washing and dewatering allowed only 25% of the cycle for cake formation. In the final calculation, a scale-up factor must be included to allow for deviation between leaf-tests and full-scale results, due largely to gradual blinding during the life of the filter medium. The exact value of this factor varies, depending upon the nature of the solids and the type of cake discharge, but most values lie between 0·7 and 0·8 with the majority being the higher value. Hence a reduced form of equation (5) may be stated :

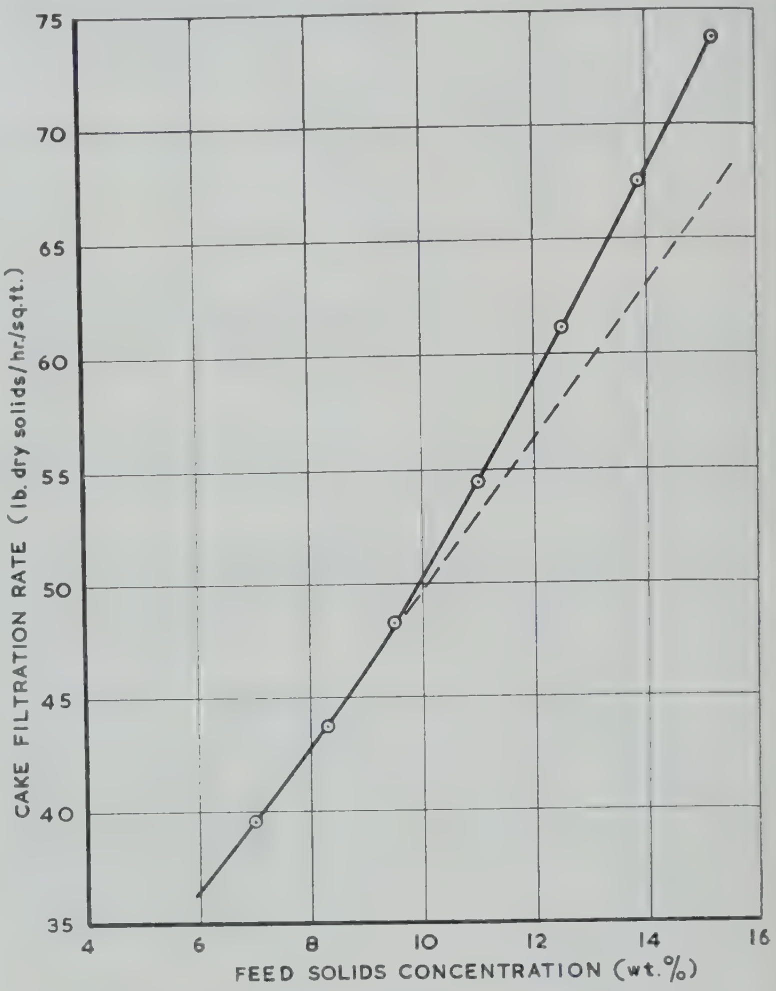
$$Y = 105\cdot3\ (\theta_c)^{-0\cdot617} \quad . \quad .. \quad (6)$$

To allow flexibility for handling poor-quality product, it was decided in this instance to base design on a $\frac{3}{4}$ in. cake. Tests had shown this could be formed in 0·75 min., equivalent to 25% of a 3 min. cycle. The design filtration rate was thus 53·4 lb. dry solids/hr. sq. ft., while the output at any other cycle time could be rapidly calculated from equation (6) and variations due to submergence could be calculated from equation (5).

Slurry concentration

Equation (4) predicts that filtrate rate is inversely proportional to $\sqrt{w}$, which is a function of slurry concentration. Similarly, cake filtration rate is directly proportional to $\sqrt{w}$. Such predictions cannot safely be made in practice, however, since α often decreases with increasing slurry concentration due to a more open cake being formed. It is therefore always advisable to investigate the influence of this factor experimentally. The importance of this is brought out in the following example, where, in fact, the decrease in α is less than occurs with many slurries.

In the extraction of uranium from ore, the uranium is obtained as a complex precipitate which is isolated by filtering and which must be washed as free as possible from soluble chloride. Washing on the filter is unreliable since the cake cracks very rapidly. Multi-stage filtration with intermediate repulping with fresh water is therefore used. The concentration to which the cake is reslurried at each stage is clearly very important since, if it is too low, the cake filtration rate will also be very low and hence the size of each filter large ; while if it is too high, a large number of stages will be needed to eliminate sufficient chloride. In investigating one such case, leaf-test data were plotted in Fig. 1, showing cake filtration rate as a function of slurry concentration : also shown is the theoretical curve calculated from the measured rate at 6·25% concentration on the assumption that α remains constant. It was then established by calculation that the optimum concentration of slurry (assuming the same concentration at each stage) was 8%, giving adequate chloride elimination in three stages.



The dotted line represents the theoretical curve from the Poiseuille equation on basis of rate of 6·26 % solids

Fig. 1.—*Filtration rate as a function of feed solids concentration*

Pressure drop

Equation (4) predicts that filtration rate is directly proportional to $\Delta P^{(i-s)/2}$, where s is the cake compressibility factor and varies from 0 to 1·0. Hence, with an incompressible cake, where $s = 0$, filtration rate is proportional to $(\Delta p)^{0\cdot5}$; and with a completely compressible cake, where $s = 1$, filtration rate is independent of ΔP. In practice, most materials lie between these extremes, but, nevertheless, a logarithmic plot of filtration rate as a function of ΔP gives a straight line relationship for which an equation may be developed similar to equation (6). Economic studies may then be made to balance increased vacuum pump or compressor costs against savings in filter costs. As the exponent of ΔP decreases, the economic pressure drop also decreases, being generally 40–60 lb./sq. in. where the exponent lies between 0·00 and 0·50.

Viscosity

Practical experience usually conforms to equation (4) which predicts filtration rate to vary inversely with the square root of filtrate viscosity. The most common way of altering viscosity is to change the temperature. Since viscosity/temperature data are either available in the literature or are readily measured, it is therefore simple to extrapolate filtration data from one temperature to another, provided no other factor, such as particle size, is also changed.

Scale-up from Cake Dewatering Rate

General background

The dewatering of filter cake depends upon the displacement of moisture by passing gas through the cake. The complexity of this phenomenon is such that no correlating equation has yet been developed but empirical methods are available. The major factors known to influence final cake moisture content are :

(1) cake dewatering time,
(2) pressure drop both during cake formation and cake dewatering,
(3) gas flow-rate through the cake,
(4) filtrate viscosity (which is sensitive to temperature changes),
(5) cake thickness,
(6) specific permeability of the cake,
(7) cake-cracking tendencies,
(8) surface properties of the solids,
(9) particle size distribution of the solids,
(10) concentration of solids in feed slurry,
(11) homogeneity of the cake,
(12) filter medium, and
(13) type and hydraulic design of the filter.

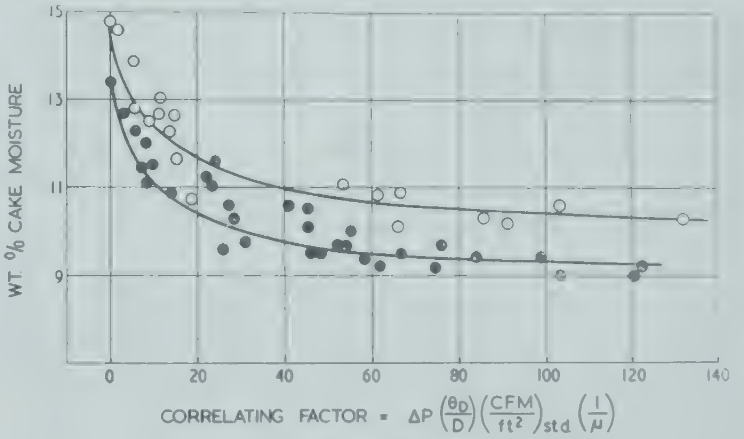
Attention can generally be limited to the first five of these factors. The other eight are either constant or, where they vary, are best treated as parameters. This is particularly true in scaling-up from leaf-test data when the type and hydraulic design of the final filter may be critical. For example, the blow-back of filtrate which often occurs when using the normal method of cake discharge on a vacuum drum filter can completely upset calculations ; this trouble may be avoided by an air purge system, string discharge, or roller discharge. Similarly, a non-homogeneous cake may be formed if the wrong type of filter is selected. By contrast, however, the first five factors are all variables which can generally be controlled by the plant operator who must therefore know their optimum values.

Principle of method

Investigation of these five factors is complicated by their interaction which prevents their being varied one at a time. A method has been devised, however, to combine the five variables into a single correlating factor of the following form[1, 9, 13, 15, 18, 19] :

$$\text{correlating factor} = \Delta P \left(\frac{\theta_D}{D}\right)\left(\frac{CFM}{A}\right)\left(\frac{1}{\mu}\right) \quad . \qquad (7)$$

From investigation of many different materials, it has been found that a plot of final cake moisture content as a function of this factor gives a smooth curve such as that in Fig. 2. The asymptotic shape is characteristic, the rate of approach to minimum moisture content increasing with cake permeability. The data plotted in Fig. 2 were obtained from leaf tests on taconite iron-ore concentrates but it is interesting to note that data from the full-scale plant deviated from the average curve by a maximum of only 0·8 percentage points.



Laboratory leaf-test data only
Filter media Saran 601
○ = with dust. –83%—325 mesh
● = no dust. 88–91%—325 mesh

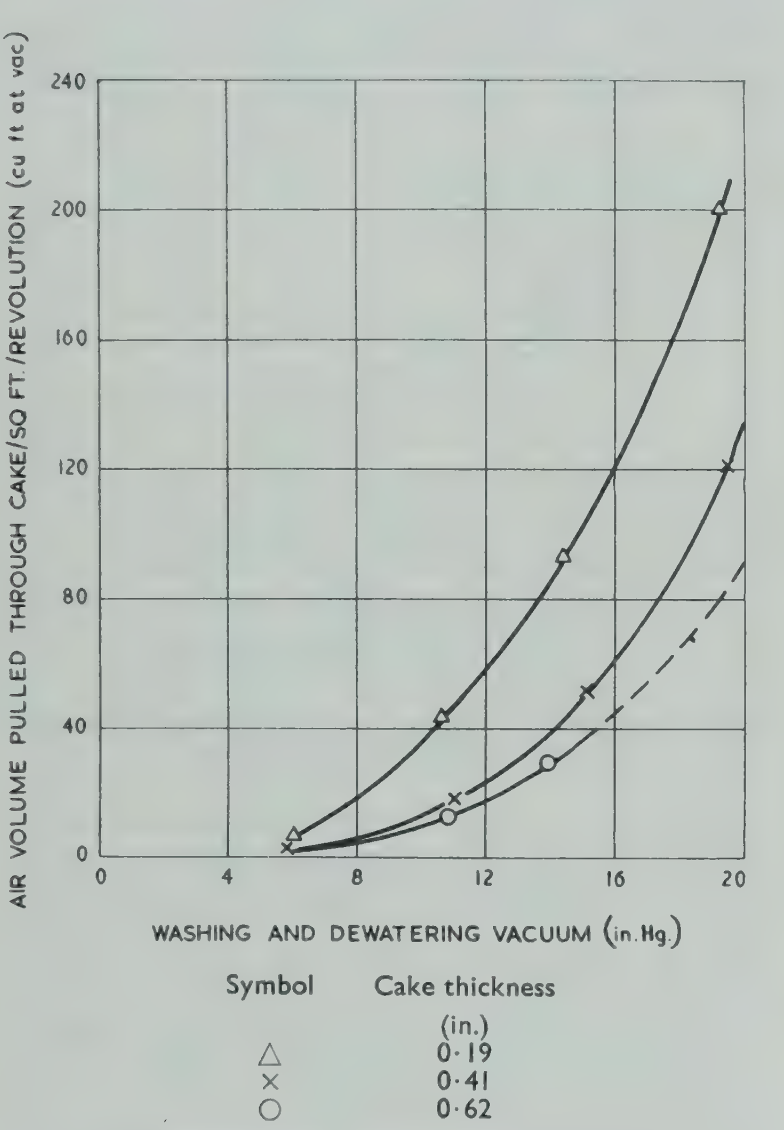
Fig. 2.—*Dewatering of taconite iron ore concentrates. Cake moisture against general correlating factor*

It will be seen that plant design should be based on a value of the correlating factor to the right of the point of inflexion of the average curve so that plant upsets will have only a slight affect upon cake moisture content. On the other hand, basing design on too high a value will cause a useless increase in capital and running costs.

Scale-up method

From the value of the correlating factor thus determined, the size of both filter and vacuum pump (or compressor) can be calculated. Thus, the cake thickness D is a measure of the weight of solids discharged per unit area per cycle. The cycle time is given by the drying time θ_D divided by the fraction of the cycle which is available for drying, this being a characteristic of the type of filter being considered. Hence the filtration rate, expressed in output per unit area per hour, can be calculated ; a scale-up factor of 0·8 should be applied to this value.

The vacuum pump or compressor requirements are similarly specified by the pressure drop, ΔP, and the gas flow rate per unit area (cu. ft./min. sq. ft.). The product of these two factors can be calculated from the correlating factor. To determine their separate values, however, a plot must be made of gas flow rate as a function of ΔP with parameters of cake thickness, since gas flow rate varies as 1/D. Fig. 3, being data from an investigation of calcium citrate, is an example of this. It is emphasised that the gas flow rate is the average over the drying period.

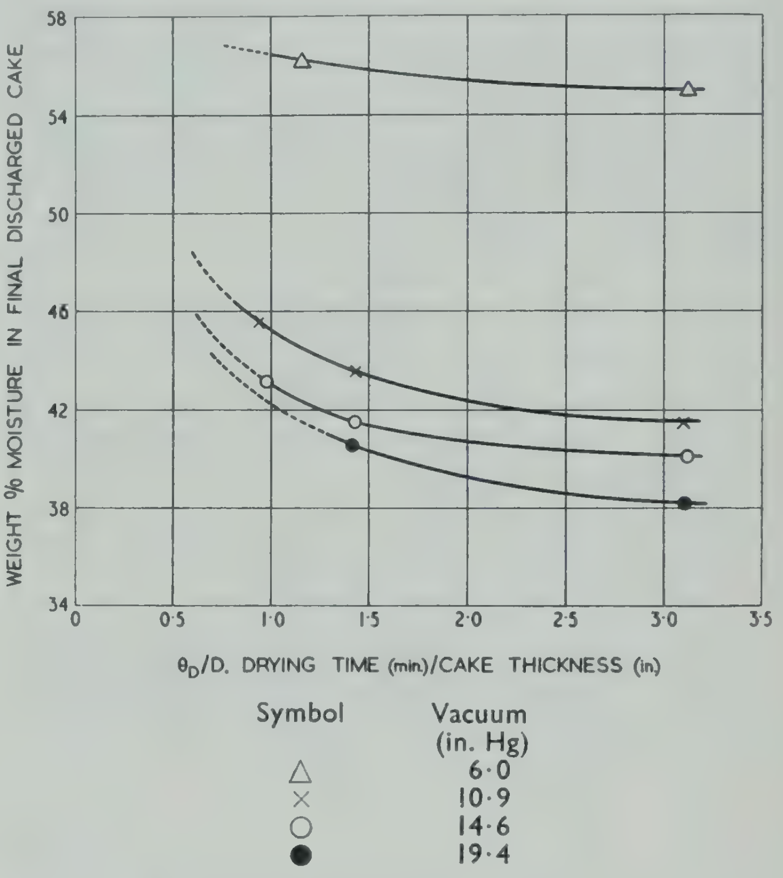


Room temperature
Initial dewatering, washing, and final dewatering time = 2·25 min.

Fig. 3.—*Filtration of calcium sulphate from aqueous citric acid*

Abbreviated forms of the correlating factor may be used where one or more of the terms is constant. For example, the temperature and therefore the viscosity are usually constant ; it is seldom economic to raise the temperature deliberately except in the case of organic liquids. Further, since the gas flow rate is a function of ΔP and D, a different form of graph may be used, cake moisture content being plotted as a function of θ_D/D with parameters of ΔP. An example of this is Fig. 4 which was plotted from data obtained during the investigation of calcium citrate referred to above.

Even further simplification is possible where the cake permeability is such that the gas flow rate is less than 3 cu. ft./min. sq. ft. and where a vacuum filter is being considered. The maximum practical operating vacuum of 23–25 in. Hg will then usually be justified so that cake moisture content may be plotted as a function of dewatering time at parameters of cake thickness.



Room temperature
Cake thickness range : 0·19 to 0·62 in.
Maximum dewatering time : 0·60 min.

Fig. 4.—*Filtration of calcium sulphate from aqueous citric acid*

Scale-up from Cake Washing Rates

Two factors determine the rate of cake washing. The first is the quantity of wash liquor required to obtain a cake of acceptable purity ; the second is the rate at which the wash liquor can flow through the cake.

Washing efficiency

A relation between the quantity of wash liquor used and the amount of soluble salt removed from a filter cake has been developed theoretically on the assumption that the process depends upon the diffusion of the wash fluid[4, 14]:

$$R = (1 - E/100)^n \quad . \quad . \quad (8)$$

R is the quantity of soluble salts remaining in the washed cake expressed as a fraction of the quantity present before washing ; n, which is termed the " wash ratio ", is the ratio of the volume of wash liquor used to the volume of mother liquor present in the cake before washing ; and E is the washing efficiency which normally lies between 35% and 86%, the more permeable cakes generally having the lower values probably because of channelling and incomplete mixing.

If equation (8) is valid, a semi-logarithmic plot of R as a function of n should yield a straight line with $R = 1 \cdot 0$ at $n = 0$. In practice, data frequently conform with such a plot. In some instances, however, a curve is obtained indicating that some other mechanism is controlling, such as desorption. Even in such cases, however, it is frequently possible to draw a reasonable straight line which errs on the safe side and can therefore be confidently used to interpolate data ; such a line is accordingly referred to as a " conservative average ".

Flow rate of wash liquor

Since no further solids are deposited on the cake during washing, it would be expected that the instantaneous flow rate of wash liquor would be constant and would equal the final instantaneous filtration rate at the end of the cake-forming period. From equation (1) the wash liquor flow rate is therefore

$$\left(\frac{dV}{Ad\theta}\right)_w = \frac{\Delta P \,.\, A \,.}{\mu \alpha w V_f} \quad . \quad . \quad (9)$$

By proper substitution, it can be shown that[4]

$$\theta_w = K'\theta_f n \quad . \quad . \quad (10)$$

In developing equation (10), it was assumed that the pressure drop during washing was equal to that during cake forming ; and that the viscosity of the wash liquor was equal to that of the main filtrate. Where the two viscosities differ appreciably, equation (9) can be corrected by multiplying by the reciprocal ratio of the viscosities provided the two liquids are easily miscible.

Equation (10) states that a plot of required wash time as a function of wash rates should yield straight line parameters for cake formation time, all passing through the origin. Furthermore, at the same wash ratio, required wash times should be directly proportional to cake-formation-times ; thus, altering the speed of a drum filter will not greatly alter cake purity provided an adequate supply of wash liquor is available.

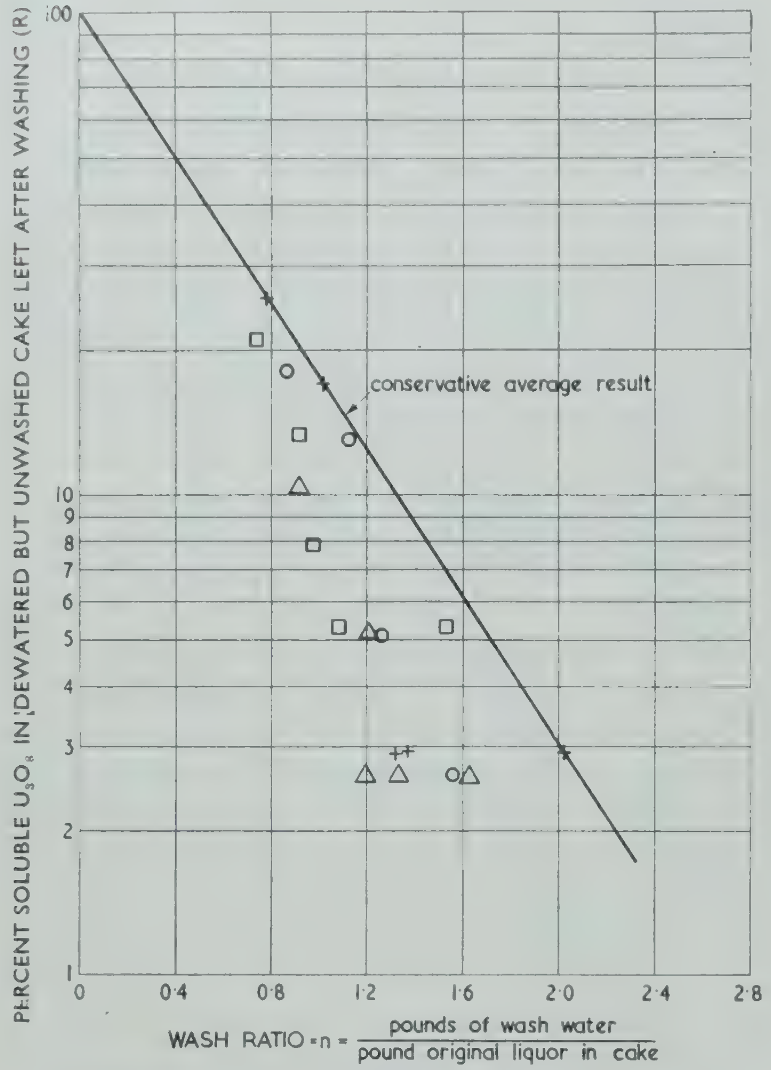
Example of scale-up from cake-washing rate

A uranium ore, ground to 55% —200 mesh, was leached with sulphuric acid and an oxidising agent at about 60% solids for 48 hr. After leaching, the " pregnant liquor " containing the uranium in solution had to be separated from very large amount of gangue solids before further processing to obtain a dried uranium precipitate. The initial uranium content might be as low as 0·08% so that an overall recovery of 99·5% of the soluble uranium present was required. On the other hand, the volume of liquor had to be restricted to prevent excessive capital and operating costs. These objectives can best be achieved by using a several-stage filter station, with intermediate reslurrying of the cake on a counter current system, and with spray-washing of the cake on each filter. Leaf-tests were therefore performed to determine washing efficiency and cake washing rates to permit calculation of the most economical filter station.

It was first determined that the influence of cake-formation-time on filtration rate yielded an exponent value of $-0 \cdot 50$ as would be predicted from equation (4) giving the reduced equation

$$Y = 505\ B\ (\theta_f)^{-0 \cdot 50} \text{ lb. dry solids/hr. form time sq. ft.} \quad (11)$$

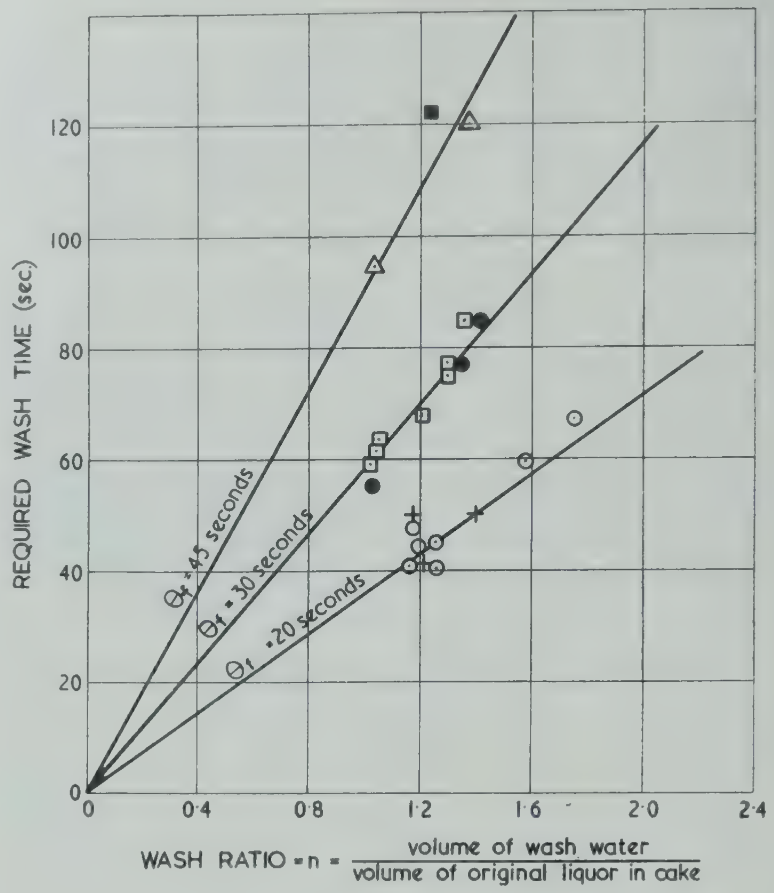
where θ_f is measured in minutes. Washing tests were then carried out and the data plotted on the basis of equation (8) to give Fig. 5. The washing efficiency, E, was found to be 82·5%, agreeing well with washing efficiency values experienced for many leached uranium ores which fall within the narrow range of 80–86%. For full-scale predictions, an efficiency of 70% was assumed to allow for uneven cake thickness and wash-water distribution. This safety margin is slightly greater than is needed for drum filters, but the 70% wash efficiency has become standard in uranium recovery calculations in order to guarantee high



Feed solids concentration range : 56·5 to 59·0 wt. %

Symbol	Batch	pH	Leach time (hr.)
○	1	2·0	48
□	2	2·0	48
△	3	1·5	25
+	4	1·5	26

Fig. 5.—*% soluble uranium in dewatered but unwashed cake remaining after washing against wash ratio*



Symbol	Stage no.	Form-time (sec.)
○	1	20
□	1	30
△	1	45
+	2	20
●	2	30
■	3	45

Filter media: cotton ST 19. Vacuum: 23 in. Hg
Leaf area: 0·1 sq. ft. pH: 1·975–1·99
Average % 200 mesh in filter cake: 55·1 wt. %
Feed % solids: Stage 1: 58·4%
Stage 2: 60·0%

Fig. 6.—*Wash time as a function of wash ratio with parameters of form-time. First- and second-stage acid leached uranium ore filters*

recoveries in the full-scale plant. Hence for this example, equation (8) reduces to

$$R = (0\cdot3)^n \qquad . \qquad . \qquad (12)$$

From the wash-liquor volumes and the moisture contents of unwashed cakes, the wash ratios were calculated. On the basis of equation (10) the observed wash times were then plotted as a function of wash ratio at parameters of cake-forming-time to give Fig. 6. The data agree well with straight lines drawn through the origin. Moreover, the ratio between the parameters at the same wash ratio is only slightly greater than the ratio of form times, thus confirming equation (10) ; the slight increase over the theoretical ratios is probably due to the slightly greater cake liquor contents prior to washing which occurred as cake formation time increased.

It is now possible to calculate full-scale filtration rate as a function of wash ratio by using equation (11) and Fig. 6. Bottom-fed drum filters are used for this process allowing 105° of the perimeter to be used for cake washing. A scale factor of 0·8 is applied to leaf-test filtration rates. To illustrate the calculation of full-scale results the following example is given :

TO FIND

Full-scale filtration rate at 0·75 min. form-time and a 1·5 wash ratio.

Wash time = 135·7 sec. = 2·26 min. (from Fig. 6).

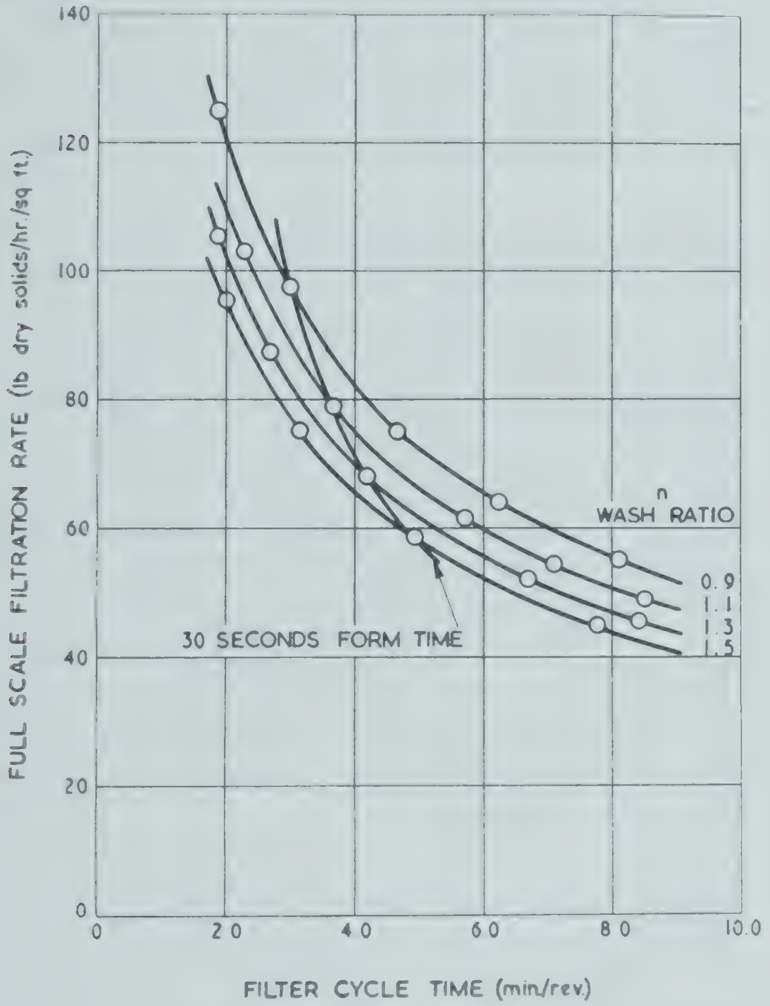
Therefore, cycle time = 2·26 × 360°/105° = 7·75 min.

Full-scale filtration rate

$$= Y = 505 \times 0{\cdot}75/7{\cdot}75 \times (0{\cdot}75)^{-0{\cdot}50} \times 0{\cdot}8$$

$$= 45{\cdot}1 \text{ lb. dry solids/hr. sq. ft.}$$

Similarly, the predicted full-scale filtration rate has been plotted as a function of cycle time at parameters of wash ratio in Fig. 7. Also included in the graph is a parameter of 30 sec. form-time which would allow a 0·5 in. thick cake to be formed; this is a reasonable design basis since approximately 30% excess capacity is then available merely by increasing the speed of rotation of the filter.



Filter media: cotton ST 19. Vacuum: 23 in. Hg
60% feed solids concentration to both stages
55% —200 mesh
80% design factor on leaf-test data

Fig. 7.—*Full-scale filtration rate against cycle time-parameters of wash ratio. Two-stage drum filtration with cake washing. Leached uranium ore slurry*

This emphasises the importance of cake-washing rates in designing full-scale filters. In the sample calculation, the 0·75 min. cake-forming-time represents only 9·7% of the 7·75 min. cycle. If washing were not performed maximum cake-formation-time would be about 35% of the total cycle time for a standard low submergence filter. Thus the necessary wash ratio for proper uranium recovery would not be achieved if design were based solely on cake-formation-time.

The full-scale recovery of soluble uranium may now be calculated as a function of wash ratio. After investigation, two-stage filtration was found to be the most economic and only details of this are given. The following assumptions, all determined from experimental data, are necessary:

(1) feed solids concentration to both filter stages = 60·0%,

(2) moisture content prior to washing = 19·7% for stage 1 and 19·35% for stage 2,

(3) moisture content of cake at discharge = 18·0% for stage 1 and 17·5% for stage 2.

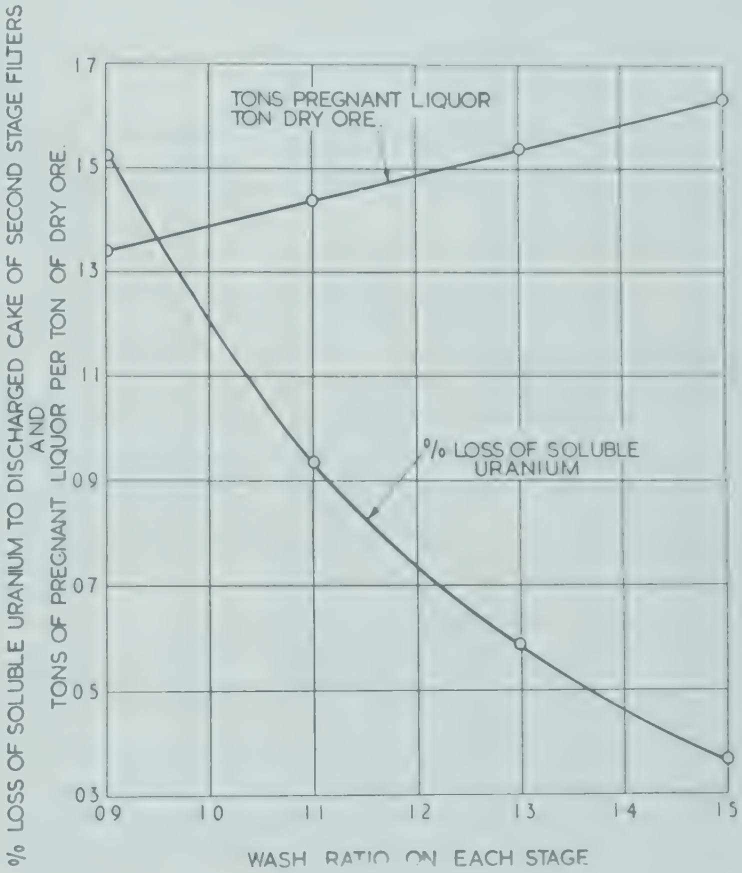


Fig. 8.—*Tons of pregnant liquor per ton of dry ore as a function of wash ratio per filter stage. Two-stage drum filtration with cake washing of leached ore. Bases as given in text*

Soluble uranium lost was then readily calculated by carrying out a material balance and using equation (12). Hence, the percentage loss of soluble uranium in the discharging cake has been plotted as a function of wash ratio in Fig. 8, assuming that the same wash ratio was used on each stage as is normal in uranium ore processing. It will be seen that a wash ratio of at least 1·363 would be required to keep soluble uranium loss to 0·5%; for safety a value of 1·5 was selected for design. For convenience a plot of tons of pregnant liquor per ton of dry solids as a function of wash ratio has also been included in Fig. 8; from this it can be seen that a wash ratio of 1·5 corresponds to 1·63 tons of pregnant liquor per ton of dry solids, which was just below the specified maximum value of 1·65 tons.

The final design conditions and predicted operating results can now be summarised:

(1) two-stage drum filtration with cake washing,

(2) design wash ratio at each stage = 1·5,

(3) design cake-formation-time = 30 sec.,

(4) full-scale design filtration rate = 58·5 lb. dry solids/hr. sq. ft. for each filter stage (from Fig. 7),

(5) design filter cycle time = 4·90 min. per revolution (from Fig. 7),

(6) tons of pregnant liquor per ton of dry ore = 1·63 (from Fig. 8),

(7) percent loss of soluble uranium in discharged cake of second-stage filters = 0·36% (from Fig. 8).

Scale-up for Thermal Drying Rate

General background

It is sometimes possible to obtain essentially dry cake from a filter by drawing hot gas through it. The method is only suited to relatively coarse solids, such as crystalline salt and ammonium sulphate, since the necessary volume of gas is very large. Such solids generally filter and settle rapidly and must therefore be handled on top-feed drum, pan, horizontal, or belt filters; thermal drying is usually restricted to the top-feed drum filter. Where the method is applicable, both capital and running costs are much lower than a combination of a filter and a separate drier, particularly since thermal efficiency is high. A further advantage is that dust problems are avoided. The unit must be very carefully sized since it is non-self-regulating. For example, if an upset in the process gives a smaller crystal size, the cake moisture content will increase thus requiring an increase in heat supply; but the cake permeability, and therefore the air flow rate, will decrease, thus causing a fall in heat supply. It is therefore advised that an excess capacity of 15–25% should be allowed in sizing the equipment.

It is rarely economic to operate these units at vacua higher than 6 in. Hg, 3 in. Hg being the optimum for many coarser materials such as the salt considered in the example below. The pressure loss through the system is liable to be high due to the very high gas flow rates but proper hydraulic design can restrict it to less than 1 in. Hg; the appropriate exhausters are therefore usually rated at 4–7 in. Hg. There is an obvious advantage in using the highest possible inlet gas temperature. Apart from possible damage to the cake, however, too high a temperature will form a crust which restricts gas flow and therefore hinders effective drying.

Principle of method

Materials suitable for handling on a filter drier usually dewater so rapidly that only a few seconds are required for cake formation, whether the cake thickness is 1 in. or 2 in. It is therefore normally considered that 75% of the cycle is devoted to cake drying, the remaining 25% being shared between cake formation and cake discharge. Design must be accordingly based on the time required to dry the cake. If leaf-tests are carried out using a constant cake formation time of say 5 sec. but varying the cake thickness, it is found that, as would be expected, cake moisture content increases with thickness, a co-ordinate plot of the data giving a smooth curve. By using the minimum cake thickness, the heat required for drying will therefore also be a minimum. Moreover, the gas flow rate will increase as cake thickness decreases, thus further reducing the required filter area. Practical considerations, however, prevent the use of a cake less than ½ in. thick and design is accordingly usually based on a ½ in. to 1 in. cake and a drum speed of 1·75–2·25 m.p.r. which allows ample spare capacity to handle off-standard products.

The cake moisture content after the initial dewatering being known, the required heat load can be calculated by a thermal balance. From measurements of the rate of flow of gas per unit area of filter through a cake of given thickness, and by assuming an inlet gas temperature, the time needed to pass the required heat load through the cake can be calculated and from this the cycle time is determined. The output of dry solids per unit area of filter can then be calculated from the cycle time, the cake thickness, and its bulk density. The size of filter required to handle a specific quantity of dry solid may then be determined and from this the total capacity of the gas exhausted calculated.

Theory

The above calculation cannot be carried out until the flow rate of the gas has been expressed in some form which will take account of the changes in density and velocity within the cake, due to the gas being cooled, moisture evaporated, and pressure reduced. These changes will vary with the inlet gas temperature. Gas velocity can be expressed as volume of gas per min. per unit area of filter, but this will not be constant. From fluid-flow theory, it would be expected that the log mean of the inlet and outlet gas rates expressed on this basis would be a function of the cake thickness and of the pressure drop across the cake; on the other hand, it should be independent of the actual conditions, such as density and pressure, at either the inlet or the outlet. Nor should the affect of temperature on the absolute gas viscosity be significant since the flow conditions must be at least partially turbulent, due to the very high gas rates required for drying.

If the above deductions are true, the log mean rate should be independent of the inlet gas temperature. The validity of this has been established from leaf-tests on sodium chloride crystals using different air temperatures, vacuum levels, cake thicknesses, and crystal-size distributions. Logarithmic plots of log mean air as a function of vacuum level gave parameters of cake thickness of slopes +0·65 to +0·9, regardless of the air temperature. The different slopes corresponded to different crystal size distributions, the finer crystals giving higher slopes; it is evident that in all cases turbulence was incomplete even though air rates were above 50 cu. ft./min. sq. ft. Similar plots of cake thickness gave parameters of vacuum level of slopes −0·65 to −0·90, again with no deviation due to temperature variations. It is therefore considered reasonable to use the log mean volumetric gas rate in carrying out calculations.

Example of scale-up from thermal drying rate

An investigation was carried out on sodium chloride crystals of the size analysis given in Table I.

TABLE I.—*Size Analysis of NaCl Crystals*

Mesh size, U.S. standard	Wt. %
+20	4·2
−20+30	39·1
−30+40	41·1
−40+50	12·3
−50+60	1·8
−60+70	0·9
−70	0·6

From leaf-test data, the dewatered cake moisture content prior to drying has been plotted as a function of cake thickness in Fig. 9 and the log mean air rate has been plotted as a function of cake thickness, at parameters of vacuum level in Fig. 10. The following assumptions were made:

(1) inlet gas temperature = 450° F.,

(2) final cake moisture content = 0·5% allowing evaporative cooling to give a bone dry cake,

(3) inlet air humidity = 0·01 lb. water/lb. bone dry air,

(4) inlet slurry temperature = 120° F.,

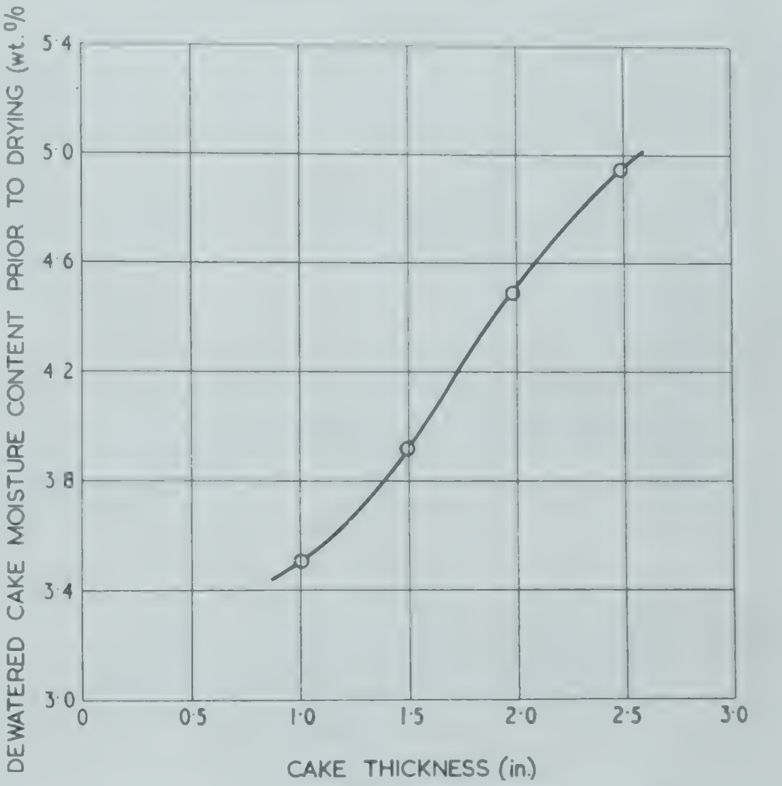


Fig. 9.—*Dewatered cake moisture content prior to drying as a function of cake thickness. Top feed thermal drying of sodium chloride crystals. Size distribution given in Table I*

(5) final discharged salt temperature = 220° F.,

(6) air leaves filter at 10° F. above adiabatic saturation temperature = 130° F.,

(7) specific heat of inlet air = 0·25 B.t.u./lb. ° F.,

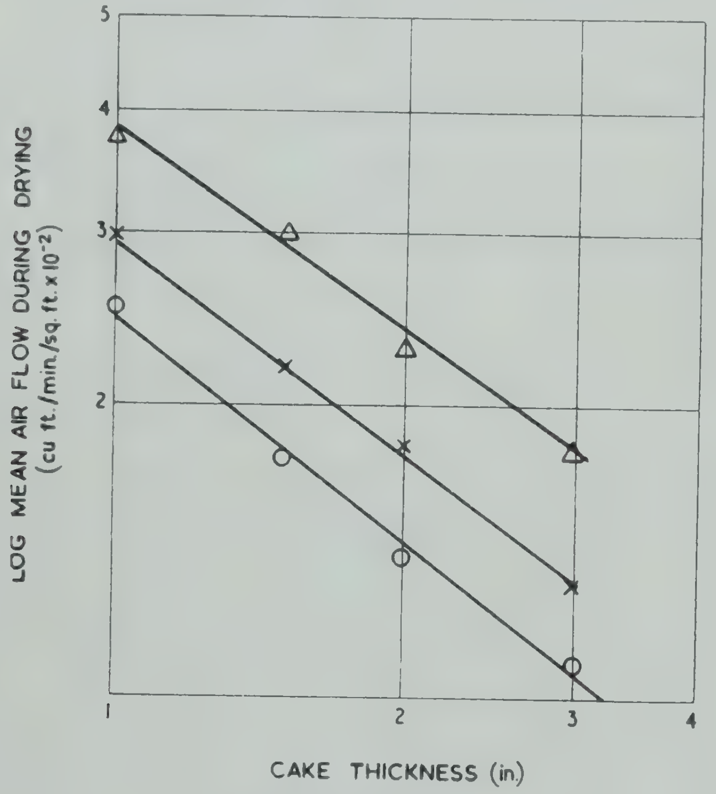
(8) specific heat of salt = 0·21 B.t.u./lb. ° F.,

(9) radiation losses, etc. = 10%,

(10) density of dry salt = 79·4 lb./cu. ft., and

(11) cake thickness = 1·5 in.

A $1\frac{1}{2}$ in. cake at a density of 79·4 lb./cu. ft. is equivalent to 9·91 lb. of salt/sq. ft. cycle. The moisture content being 3·92% (Fig. 9) 0·354 lb. of water need to be evaporated per sq. ft. cycle, requiring 364-B.t.u./sq. ft. cycle. The salt will absorb 209 B.t.u./sq. ft. cycle in being heated to 220° F. Allowing 10% radiation losses, the total heat required is 636 B.t.u./sq. ft. cycle. Hence, 7·95 lb. of inlet air are required 1 sq. ft. cycle, equal to 181·8 cu. ft. at 450° F. and atmospheric pressure. The discharging air plus the evaporated water is equal to 0·299 moles/sq. ft. cycle, which is 143 cu. ft. at 130° F. and 3 in. Hg vacuum. Thus, the log mean air volume is 162·1 cu. ft. From Fig. 10, the log mean air rate for a 1·5 in. cake at 3 in. Hg vacuum is 179 cu. ft./min. sq. ft.; the required drying time is therefore 0·906 min.



NaCl size distribution

U.S. Mesh standard	Wt. %
+20	4·2
20×30	39·1
30×40	41·1
40×50	12·3
50×60	1·2
60×70	0·9
−70	0·6

Symbol	Vacuum level (in. Hg)	Slope of straight line
△	6	−0·69
×	4	−0·72
○	3	−0·77

Fig. 10.—*Log mean air rate during drying against cake thickness parameters of vacuum level. Top feed dryer leaf-tests on sodium chloride crystal*

Since 75% of the cycle is devoted to drying the cycle time is 1·21 min. Thus 9·91 lb. of dry salt/sq. ft. cycle is equal to 491 lb./sq. ft. hr., from which the filter size may be calculated. The exhauster size is calculated from the outlet gas rate given above as 143 cu. ft./sq. ft. cycle measured at 130° F. and 3 in. Hg; this is equivalent to 122·7 cu. ft./min. sq. ft. at 130° F. and 4 in. Hg. The heat requirements given above as 636 B.t.u./sq. ft. cycle, are similarly equivalent to 624 B.t.u./cu. ft. min. or 76·3 B.t.u./lb. of dry salt.

Symbols Used

A = area.

D = cake thickness.

E = wash efficiency % = $(1 - R)$ 100 at $n = 1 \cdot 0$.

K, K' = proportionality constants.

n = wash ratio $= \dfrac{\text{volume of wash fluid}}{\text{unit volume of liquor in cake prior to wash.}}$

R = weight fraction of dissolved salt or strong liquor remaining in the cake after washing on the basis of $R = 1 \cdot 0$ prior to washing (*i.e.*, if no washing is performed, $R = 1 \cdot 0$).

r = resistance of unit area of filter media plus internal piping, etc.

s = cake compressibility exponent on Δp ranging from 0·0 for non-compressible cakes to 1·0 for highly compressible cakes.

V = volume of filtrate.

V_f = volume of filtrate at θ_f.

w = weight of dry cake solids per unit volume of filtrate.

Y = filtration rate expressed as $\dfrac{\text{volume filtrate}}{\text{hr.} \times \text{unit area}}$ or $\dfrac{\text{weight dry cake solids}}{\text{hr.} \times \text{unit area}}$.

(CFM) = standard cu. ft. of gas per min. measured at atmospheric pressure and 60° F.

$\left(\dfrac{dV}{Ad\theta}\right)_w$ = wash fluid rate expressed as volume per unit time per unit area.

α = specific cake resistance.

α' = specific cake resistance at $\Delta p = 1 \cdot 0$. Derived from $\alpha = \alpha'(\Delta p)^s$.

Δp = pressure drop across filter.

θ = time.

θ_D = cake dewatering time.

θ_f = cake formation time.

θ_w = cake washing time.

μ = filtrate absolute viscosity.

Unless otherwise specified, any consistent system of units may be employed for nomenclature.

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DISCUSSION OF PAPERS PRESENTED AT THE FOURTH SESSION

Mr. H. Hoog said that the Shell organisation had a catalytic desulphurisation process, and it was appropriate to refer to some of the parallels and some of the divergencies between the autofining process and the Shell desulphurisation processes, particularly as he could emphasise differences in approach.

In Shell they had not gone to the trouble to which British Petroleum had gone in trying to develop a process that would be self-sustaining in hydrogen. They started from the assumption that there would be extraneous hydrogen for the hydrogenation of the sulphur compounds. They looked for a selective catalyst, and they too had come to this curious combination of mixed cobalt and molybdenum oxides on alumina, which was an extremely selective catalyst and took sulphur out without touching the aromatics to any extent. After that, however, their approaches differed. In the Shell process they used extraneous hydrogen and as low a temperature as possible and a somewhat higher pressure than in autofining. Under those conditions they obtained very good selectivity in sulphur removal.

Next, to prolong the catalyst life, they thought that it would be an advantage to keep the catalyst in a continuous phase of hydrogen, and instead of passing a mixture of hydrogen and reactant vapour over the catalyst they decided to have a continuous atmosphere of hydrogen and reactant liquid trickling down over the catalyst. That was why they had called the process trickle desulphurisation.

With regard to the various scales of plant for development, the natural thing to do was first to use a bench-scale unit and try out the idea on a laboratory scale, and so they had begun with laboratory units of small size, 50–200 ml., and then went to the pilot-plant scale, with hydrogen recycling and all the additional apparatus which had to be brought in. Their largest pilot-plant had a 3 in. diam. tube and a total height of catalyst of about 15 ft. The reactor had a volume of about 20 l. reaction space. This, the largest pilot-plant built at Amsterdam, had a capacity of 6 barrels a day. That showed a distinct difference in the approach; they did not go beyond 6 barrels a day, compared with 100 barrels a day pilot plant used by British Petroleum. From that size, however, they had scaled-up directly, with the help of the design staff at The Hague, to a unit with a capacity of 7000 barrels a day, and which now, with only some additional pumps the replacement of a few burners in the furnace, had a capacity of 10 000 barrels a day. That meant a scaling-up factor of 1000.

That had been possible only by a series of experiments with models. They had done model work on the flow of liquid down to the catalyst beds, the liquid distribution over the catalyst beds; they had studied the wall effects and the effect of the size of the catalyst pellets so far as flow pattern was concerned. They studied the pattern in heat-exchangers where there were liquids and gas together, and the heat transfer coefficient for a liquid rising through a heat exchanger together with a gas. In this work they had found[1] that the heat transfer coefficient was raised very considerably as compared with that obtaining when only a liquid was passing through the tubes. All those principles had been worked out on a model scale and not with the actual reactors. This had meant a great saving in money and might be an example which fitted into the general pattern of the discussions of the last two days.

The scaling-up from the 6 barrels a day pilot-plant also required study of the cooling of the future commercial reactor in which it had been intended to do this by injecting cold feed between the beds. This required the study of a mixing tray for hot oil coming down from a catalyst bed with the cold feed in between. That had been done with water and a coloured liquid on a scale of about two-thirds the ultimate commercial size.

By this general "model-approach" they had solved the basic chemical engineering problems connected with the ultimate scaling-up.

With regard to the materials of construction, the reactor required anti-corrosive material and it had a high chromium lining, but outside they did not have the same problems as the authors. They removed hydrogen sulphide from the recycled gas and they could therefore use low-chrome steel tubes without difficulty. After a year's operation they had had no real corrosion and no scale trouble on the catalyst, and on the whole they were quite happy about their process. This was now in operation on a commercial scale in three Shell refineries and, under licence, in two others, while ten more plants were being built.

Prof. M. W. **Thring** said that Dr. Bacon's paper was an example of what might be called empirical similarity. He tried to develop a theory for similarity—the equivalent nozzle diameter theory—but because the full-size plant was already operating, it was possible to check the results obtained with the model. That was different from the usual case, where it was necessary to predict how a full-size plant would go from a pilot plant, and it had the great advantage that, as Dr. Bacon mentioned, it had been possible to make sure that the similarity was right in one case by taking detailed measurements.

Dr. Bacon referred to the efficiency of the nozzle in terms of total heat. Prof. Thring did not think that that was a good way to define the efficiency of a nozzle using oil as well as steam, because the total heat was a little uncertain. He suggested that the right way to define the efficiency of the nozzle was the momentum production efficiency, the relationship of the momentum actually obtained to that which might be got ideally, because the jet momentum was the criterion of a good burner. On the other hand, several workers tended to regard jet momentum as all-important, although it was not the jet momentum which mattered but the ratio of the mass input rate of fuel to the square root of the jet momentum. That was why he was glad that Dr. Bacon mentioned the equivalent nozzle diameter, because that governed the effective flame length if there were a free jet, and it was upon this that interest was centred.

Another case of empirical similarity was the heating of coke-oven flues by coke-oven gas, where the problem was to get a very long flame which would not finish burning until the top. The similarity conditions there were different. There was a very low Reynolds number the value of which was exceedingly important, so that it was necessary to have the right Reynolds number in the gas nozzle, the air nozzle, and the final combustion chamber. That involved cheating with the dimensions, adjusting them in the opposite sense to those of Dr. Bacon, who had an enlarged nozzle; the actual nozzles were smaller than in geometrical proportion. There again, however, it was possible to test the similarity by putting a probe into a coke-oven flue to see whether the right result was obtained in one case, and having done that one could make changes in the model and get a system which one hoped would behave better than the real one and then try it. That was the basis of this furnace similarity, which was used to so large an extent.

This same concept of the equivalent nozzle diameter ought to be applied to all the work which was being done on water models and air models of gas turbine combustion chambers, because exactly the same considerations applied; *i.e.* there was in essence a cold air jet penetrating into a hot gas stream, and therefore if an isothermal model was to be made it ought to have the enlarged nozzle which would give the correct jet momentum for a given mass flow.

Dr. F. SJENITZER said there were four points to which he wished to draw attention in the paper by Dr. Dahlstrom and Mr. Purchas. The first was the theoretical background. On p. S121 the usual assumption was made that the resistance of the filter cloth was negligible, although the authors realised that this was not always true. For conditions where this assumption could not be made, he would like to refer to work by Ruth and Kempe,[2] Mondria,[3] and Sjenitzer.[4] In these papers fairly simple equations were given in which the resistance of the filter cake and the filter cloth were both taken into account. For the notation of the present paper the complete equation would read:

$$\frac{V_f}{A} = -\frac{r}{\alpha w} + \sqrt{\frac{r^2}{\alpha^2 w^2} + \frac{2(\Delta p)^{1-s}\,\theta_f}{\mu\alpha' w}} \quad . \qquad (3a)$$

For small values of r, this reduced to equation (3), p. S121.

On p. S122, under the sub-heading "Slurry concentration", in Fig. 1 a curve was given in which the cake filtration rate was plotted as a function of the feed solids concentration. When the correct co-ordinate axes were drawn—(*i.e.* for concentration = 0 and filter rate = 0)—it was found that the curves giving the relation between rate and concentration had rather an unexpected form. They should, of course, pass through the origin. In his own paper[4] he stated that the resistance should be as follows when the resistance of the filter cloth was negligible:

$$P = \sqrt{\frac{C.H}{1 - \epsilon - c}} \qquad \text{(equation 28)}$$

where P was the cake rate, H a constant, c the volumetric concentration of solids in slurry feed, and ϵ the volumetric porosity of the cake. This curve went through the origin. The slope became at first less steep with increasing concentration C, but later became steeper, and this checked with the points in Fig. 1 of the present paper.

His third point concerned the cake dewatering rate, equation (7), p. S123. When dimensions were substituted it appeared that the "correlating factor" was not dimensionless but had a dimension of 1/time. He had tried to get another dimensionless equation, assuming that the same factors played a role, and he found a slightly different equation :

$$\frac{D.A}{\theta_D(CFM)} = F\left(\frac{\Delta p\theta_D}{\eta}\right)$$

On the other hand, as already mentioned by the authors, particle size should be taken into account. When some characteristic particle diameter δ was introduced, it was obvious that the ratio δ/D should also play a role, and this had to be evaluated by experiments.

His fourth point was concerned with washing efficiency, p. S125. He wished to make a comparison between washing efficiencies with various materials as determined by Dahlstrom and Purchas in the present paper ; Rhodes, of Cornell University, in work referred to in the present paper ; and Sjenitzer and Mondria, in unpublished work undertaken at the Royal Dutch Shell Laboratory, Amsterdam.

As stated in the present paper, the washing efficiency E during the second period of "diffusional washing", which followed after the period of "displacement washing", lay normally between 35% and 86% for a washing liquid volume = pore volume of the filter cake. Rhodes, in 1934, reported experiments of the washing of a salt solution from a filter cake consisting of diatomic (Filter-Cel) particles. He too found that the fraction of salts not yet removed from the cake decreased exponentially with the amount of washing liquid, and from his data Mr. Sjenitzer derived E = 47% for a washing liquid volume = filter cake volume. In 1949, he and Mondria had carried out experiments on a laboratory scale on the washing of paraffin wax filter cakes. The result was, for a wash volume = 1 pore volume of the filter cake, that the efficiency E was 60–85%, depending upon the occurrence of cracks in the filter cake. In these experiments the exponential character of the removal of filtrate by washing after the period of displacement washing had also been established.

Mr. M. G. PERRY said that at the end of Dr. Bacon's paper there was a section on the premelting of scrap by a counterflow system and the study of such systems by wax models. He would like the author to indicate the possibilities, if any, of determining on a model scale the effect of varying size and shape of scrap, particularly in relation to the dimensions of the shaft. This would have an important bearing on the flow of solid material through the shaft and of course affected the packing density of the solid and so the flow distribution of the gases used in the counterflow system.

Dr. A. KLINKENBERG said that while he would not give a direct answer to Prof. Thring's question regarding the size of steps to be taken when scaling-up to the technical plant he would like to point out that there might be cases where the problem did not arise: namely where it was possible to foresee which technical factors would govern the full-size unit. If the right experiments were made on hydrodynamic and heat transfer problems to make it possible to understand how the full-scale unit would work, it should be possible to design a full-scale unit right away, without any pilot plant work.

That brought him back to a point raised by Prof. Thring and Mr. Edgeworth Johnstone in their introductory paper when they mentioned that there were cases where the process failed to work in the pilot-plant but might work perfectly satisfactorily on a large scale. They mentioned Hall's electrolytic process for the manufacture of aluminium. Dr. Klinkenberg was not familiar with the process in question and did not know whether this was a matter of foresight or of obstinacy; but there were instances of where this kind of foresight was definitely present, and mention should be made of types of very large-scale equipment which, while not exactly in the realm of chemical engineering, were nevertheless closely related, and where this kind of foresight was decidedly present. He referred to all kinds of nuclear reactors, including atomic bombs. They could not work on a small scale, because there was a minimum size below which they would not work. In those cases it was evident that, even in 1939, people knew at once that those plants could be designed on a big scale, although they could not be operated at all on a pilot-plant scale. That was an additional argument to show that the big unit could be designed.

Mr. F. J. WAGSTAFFE said that it was well known that the application of pressure to a filter cake affected the performance of the filter, and in particular, the moisture content of the cake. Had Dr. Dahlstrom and Mr. Purchas done experiments on a laboratory scale on the effect of applying pressure to a cake, and had they been able to extrapolate the results of those laboratory tests to a full-scale continuous filter of any type?

Mr. D. B. PURCHAS recalled that Prof. Thring, replying at the first session to a point made by Mr. Sjenitzer who had classified scaling into four categories, had suggested that the symposium was devoted almost entirely, if not entirely, to the second of these, namely scaling-up one small piece of equipment of a given design to a larger piece of equipment of the same design. It might be said that the methods which Dr. Dahlstrom had developed did not come within that range, but were in Mr. Sjenitzer's third category, as being applicable within certain limits to quite a wide range of different kinds of equipment.

Authors' Replies

Dr. N. P. BACON agreed with Prof. Thring that for the full-size burner with mixed oil and steam injection, the use of the definition of nozzle efficiency given on p. S103 must be viewed with suspicion. In the paper, however, this definition was applied to the nozzle used in the model and which passed air only.

Dr. Bacon thought that the distortion of the scaling-down of air inlets to allow for the fact that the full-scale system was not isothermal could be extended to almost any combustion system in which there were, downstream from the main burner, secondary air inlets, *e.g.* in many steelworks reheating furnaces.

Wax models had been used to attempt to simulate the shaft of a furnace where it was intended to put cold scrap in at the top and it was hoped that the scrap would pass down the shaft. The indications at the moment were that the scrap would not. Wax models gave the right gas flow pattern and a simple Reynolds' number relation; but before deciding from model work whether or not the steel scrap would flow, data were needed on the development of the respective adhesive forces within backed beds of wax and of steel.

Mr. F. W. B. PORTER, referring to the fact that both Mr. Hoog and Prof. Thring had mentioned the possibility of defining rules for scaling-up, said that he did not think that it was possible to lay down precise steps for scaling-up from the laboratory stage to commercial operation for the petroleum industry. There were so many factors, such as the need to provide large quantities of material for special tests, to be considered, especially with a new process. These made a fairly large pilot-plant a necessity.

In B.P.'s particular case at Grangemouth, the size of the unit had been influenced to a certain extent by the availability of equipment; the necessary furnace, compressors, and heat exchangers were available and a reactor had therefore been built to suit these. There were certain rules which were applied and which he thought were common throughout the industry. For example, the length of the pilot-plant reactor approximated to one which would be used on a large scale. The minimum diameter was usually governed by the particle size of the catalyst to be employed and a rule was employed that six to eight times the catalyst particle size should be the minimum diameter. This rule could be varied depending upon whether the materials were totally vapour phase, liquid, or partially liquid phase.

With regard to Mr. Hoog's remarks, B.P. had been able to avoid the pilot-plant stage and had developed a hydrofining process from a 2 in. reactor, with three to four litres per hour throughput, directly to the 6000 barrels per day scale, and a similar course had been taken with other processes. The decision to go through the pilot-plant stage was governed by circumstances not necessarily related to scaling-up problems.

Dr. D. A. Dahlstrom said that all the points raised by Dr. Sjenitzer were extremely important. One tried to be able to ignore filter-cloth resistance when selecting the right filter medium. In most cases it was possible to find a few filter media which gave better than the average performance, and when laboratory and pilot plant tests were made it would be found that the resistance of the filter media in most cases would be low or negligible. Even if it was appreciable, a quadratic equation would be obtained which would plot quite well on log-log paper and give a fairly straight line; the curvature would be slight, and for extrapolating to full scale it was usually within what might be called engineering accuracy.

Dr. Sjenitzer's comments on the influence of solids concentration were valuable. It was almost impossible to predict what would happen as the solids concentration was increased. They had made many graphs of filtration rate as a function of solids concentration, keeping everything else as constant as possible, but there was a wide variation in the specific cake resistance as a function of the solids concentration. It varied from material to material. It was difficult to tie it down to a simple equation, and he would hesitate very much to apply only one equation to it.

With regard to the correlating factor employed in the paper, the authors did not mean it to be dimensionless. Dr. Dahlstrom appreciated Dr. Sjenitzer's comments about making it dimensionless, but it had been developed from statistical methods and found to give the best empirical results and so had been employed. However, a paper by Nelson and Dahlstrom[5] in which they started from an equation derived by Brownell and Katz[6] which was thought to have a very good theoretical basis. It was complicated. Going on from there and making certain simplifications, they had been able to evolve an equation which they felt better represented the influence of different variables on the dewatering of a filter cake, and which was as follows:

$$\%M = f(F_a,\ \%M_r,\ y)$$

where the symbols have the meanings given on p. S136. This could be broken down to the correlating factor which had subsequently been developed, so that there was some theoretical backing for the correlating factor.

With regard to Mr. Sjenitzer's comments on washing efficiency, in some recent work the Du Pont Company had developed a different curve from that which the authors obtained, but it followed the straight line relation given in the paper for wash ratios up to $2\frac{1}{2}$. In most continuous filter applications it was not possible to depend on wash ratios beyond $2\frac{1}{2}$, and it was more economic to go to two-stage filtration than to try to go to wash ratios above $2\frac{1}{2}$ on a one-stage filter. The authors felt, therefore, that the equation was applicable to a wash ratio of about $2\frac{1}{2}$.

On the question of applying pressure to the cake and the problem of scaling-up, one did not usually apply pressure to filter cakes, because the majority of filter cakes did not show any appreciable lowering of moisture content with compression or flattening of the cake, but in some cases this did come into account and there was a certain lowering of the moisture content. The real problem in scaling-up was a mechanical one, to be able to apply the force which it was necessary to apply and still have dependable operation. In many cases better results would be obtained on the full-scale unit than on the pilot-plant, because it was difficult to put more than one compression roll on a small filter, whereas on full scale one could put on four or five. They did not feel that it was safe to scale-up on the leaf test; it was necessary to go to pilot plant operation to show what was necessary for good operation.

Symbols Used

$\% M$ = weight % moisture in filter cake.

F_a = approach factor = $\dfrac{Y_f \theta_D \Delta P}{b \Delta P^1}$.

$\% M_r$ = weight % residual moisture in filter cake.

y = exponent depending on particle size.

Y_f = form filtrate rate, volume per unit area per unit cake formation time.

θ_D = cake dewatering time.

b = weight of dry cake solids per unit area per cycle.

ΔP = pressure drop across cake during dewatering.

ΔP^1 = pressure drop across cake during cake formation.

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Concluding Proceedings

Dr. J. C. VLUGTER, the official representative of Het Koninklijk Instituut van Ingenieurs, Chemical Engineering Group, expressed the thanks of himself and his Dutch colleagues for the hospitality they had enjoyed.

Sir HUGH BEAVER, President of The Institution of Chemical Engineers, briefly reviewed the scope of the symposium and terminated the proceedings.

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